LABORATORY SEDIMENT BIOASSAY REPORT ON ST. LAWRENCE RIVER SEDIMENTS NEAR CORNWALL, ONTARIO 1997

OCTOBER 1999

MOE
LAB
AJHN



Ministry of the Environment

c.1
a aa

Laboratory Sediment Bioassay Report on St. Lawrence River Sediments near Cornwall, Ontario 1997

Prepared by:
D. Bedard
Ontario Ministry of Environment
Standards Development Branch
125 Resources Road, Etobicoke Ontario

October 1999

Cette publication technique
n'est disponible qu'en anglais.

Copyright: Queen's Printer for Ontario, 1999
This publication may be reproduced for non-commercial purposes with appropriate attribution.

Printed on 50% recycled paper
including 10% post-consumer fibre

ISBN 0-7778-7735-X

PIBS 3882E

MOE
LMB
AJHN

ACKNOWLEDGEMENTS

The author thanks Steve Petro for his valuable assistance in conducting the laboratory sediment bioassays and Ontario Ministry of Environment, Environmental Monitoring and Reporting Branch and Environment Canada for providing field support. The author gratefully acknowledges the staff of OMOE Laboratory Services Branch for providing chemical analysis of sediment and biota samples.

Special thanks to the following reviewers for their helpful comments and suggestions, Lisa Richman, OMOE Environmental Monitoring and Reporting Branch, Johann (Hans) Biberhofer, EC Environmental Conservation Branch and Dr. Brian Hickey, Raison Region Conservation Authority.

EXECUTIVE SUMMARY

Sediments in the St. Lawrence River were sampled in October 1997, as part of an assessment to characterize the potential ecological impacts of contaminated sediments for the Area of Concern. Sediment was sampled from seven sites along the Canadian shore extending from the Lamoureaux Park boat launch and downstream for a distance of two kilometres, along the Cornwall, Ontario waterfront.

An objective of this study was to assess the spatial pattern of sediment toxicity and chemical bioaccumulation using static, laboratory sediment toxicity tests. Three independent toxicity tests were performed on bulk sediment samples. Mortality, growth and avoidance behaviour of the burrowing mayfly, *Hexagenia limbata*, was measured in a 21-day test. Chironomid (*Chironomus tentans)* growth and mortality was measured in a 10-day test. Mortality and inorganic chemical uptake by the juvenile fathead minnow, *Pimephales promelas*, was examined by a standard 21-day test.

In terms of organism test response, a single sediment sample (station CS-131), elicited a significant sublethal effect using mayfly nymphs. Percent growth reduction was 47% relative to the weight obtained by the reference nymphs. Correlation analysis, examining the relationship between mayfly growth across all sites and sediment parameters, failed to identify any significant associations with known sediment chemical concentrations and only a weak response with sediment nutrient content or total organic carbon. The other endpoints including mayfly survival, midge survival, minnow survival and midge growth were similar among test and reference sediment exposures.

Sediment mercury concentrations ranged from 0.7 µg/g to 6.1 µg/g (dry weight) for the test sediments. Three of the test sediments had a total mercury concentration that surpassed the Provincial Sediment Quality Guideline Severe Effect Level concentration of 2.0 µg/g. Mercury sediment concentration was not associated with a significant biological response. Total mercury availability from sediment to the fathead minnow was variable among sites. Generally the biota-sediment accumulation factors (BSAFs), measured as the ratio of total mercury concentration in the fathead minnow to that in the bulk sediment, were higher at the less contaminated sites, including the reference animals. The whole-body tissue concentrations of mercury measured after the 21-day exposure in the test sediments were less than twice the corresponding tissue concentration measured for the control animals. The highest body burden was 0.17 µg/g total mercury on a wet weight basis at station CS-128. The corresponding dry-weight BSAF was 0.21. Minnow mercury tissue concentrations were well below the International Joint Commission Aquatic Life Hg guideline of 0.50 µg/g (wet weight) for all test sediments.

In summary, the majority of the St. Lawrence River test sediments were of a high sediment quality based on a lack of significant biological effects and the degree of mercury uptake and availability. One site was slightly impaired based on the outcome of one test endpoint. Whole-organism mercury concentrations measured on laboratory-exposed fathead minnows were comparable with those found in field-collected forage fish species. Movement of mercury-contaminated sediment into the digestive tract of bottom-feeding fish may serve as a possible route of transfer into the local aquatic food chain through predator-prey interactions.

TABLE OF CONTENTS

Acknowledgements ii
Executive Summary iii
List Of Tables v
List of Figures vi

SECTION 1 INTRODUCTION 1

SECTION 2 MATERIALS AND METHODS

2.1 Sample Collection and Site Description 2
2.2 Analytical Methods 4
2.3 Laboratory Biological Testing Methods 5
2.4 Statistical Methods 8

SECTION 3 RESULTS

3.1 Water Quality Test Parameters 8
3.2 Sediment Characterization 10
Physical and Nutrient Properties 10
Trace Metal Sediment Concentrations 12
Organic Chemical Sediment Concentrations 12
3.3 Mayfly (*Hexagenia limbata*) 21-day Lethality and Growth Results 16
3.4 Chironomid (*Chironomus tentans*) 10-day Lethality and Growth Results 16
3.5 Fathead Minnow (*Pimephales promelas*) 21-day Lethality Results 20
3.6 Quality Assurance Data 20
3.7 Chemical Bioaccumulation in *Pimephales promelas* 21

SECTION 4 DISCUSSION 26

SECTION 5 CONCLUSIONS 34

SECTION 6 REFERENCES 36

LIST OF TABLES

Table 1 1997 St. Lawrence River sampling station locations 2

Table 2 Mean water quality characteristics in 1997 sediment bioassays 9

Table 3 Sediment physical and nutrient characteristics in control and St. Lawrence River 1997 sediment used in sediment bioassays 11

Table 4 Bulk concentrations of trace metals in control and St. Lawrence River 1997 sediment used in sediment bioassays 13

Table 5 Bulk sediment concentrations for chlorinated organics and pesticides in reference and St. Lawrence River 1997 sediment used in bioassays 14

Table 6 Concentrations of selected organic compounds in St. Lawrence River 1997 sediments used in sediment bioassays 15

Table 7 Bulk concentrations of PAHs, total PCBs and TPHs in St. Lawrence River 1997 sediment 17

Table 8 Summary of biological results on mayfly, midge and minnow sediment bioassays for control and St. Lawrence River 1997 sediments 18

Table 9 Mean metal concentrations in fathead minnows exposed to control and St. Lawrence River 1997 sediments in the laboratory and associated biota-sediment accumulation factors 22

Table 10 Bulk concentrations of trace metals and TOC in reference and St. Lawrence River sediment that was stored and used in the calculation of biota-sediment accumulation factors 23

Table 11 Spearman rank correlation analysis summary indicating significant negative or positive correlation between sediment bulk metal chemistry and fathead minnow whole-organism bulk metal concentrations for St. Lawrence River 1997 reference and test sediments 25

Table 12 Spatial variability in sediment toxicity for St. Lawrence River 1997 samples . . 29

Table 13 Spearman rank correlation coefficients indicating significant positive correlations among toxicity data for St. Lawrence River 1997 sediments 30

Table 14 Spearman rank correlation analysis summary indicating significant negative or positive correlations between biological endpoints and sediment physical and chemical parameters for St. Lawrence River 1997 samples 30

LIST OF FIGURES

Figure 1 1997 St. Lawrence River sediment sampling stations 3

Figure 2 Mayfly and midge growth for St. Lawrence River 1997 sediments 19

Figure 3 Total mercury concentration in fathead minnow and sediment 27

1.0 INTRODUCTION

In the Fall of 1997, the Ontario Ministry of Environment (OMOE), Environmental Monitoring and Reporting Branch (EMRB) and Environment Canada (EC), Environmental Conservation Branch, Restoration Programs Division, undertook a study to assess sediment quality in the St. Lawrence River in support of the St. Lawrence River Remedial Action Plan (SLR-RAP). The work was completed in partnership with EC, National Water Research Institute (NWRI). The study area covered three zones of sediment deposition along the shoreline of the north channel of the St. Lawrence River, adjacent to the City of Cornwall, Ontario.

The St. Lawrence River has been identified as an Area of Concern (AOC) in the 1970's by the International Joint Commission (IJC). A requirement of the Remedial Action Plan (RAP) process is to gather information to determine the state of the environment with respect to delisting criteria set out by the Great Lakes Water Quality Agreement. A number of beneficial uses were found to be impaired of which many were directly or indirectly affected by sediment contamination. The beneficial uses included (i) restrictions on fish and wildlife consumption, (vi) degradation of benthos, and (vii) restrictions on dredging activities (SLR-RAP Team, 1992). The Stage 2 RAP report gives a complete summary of all previous studies for the Canadian portion of the AOC in fulfilment of providing necessary information to guide implementation strategies (Dreier *et al.*,1997). Field sediment surveys completed in 1991 and 1994 by Richman (1994;1996) confirmed the presence of copper, lead, zinc and mercury in excess of the Provincial Sediment Quality Guidelines Severe Effect Level (PSQG-SEL) (Persaud *et al.*, 1992), along with high sediment concentrations of oils and greases. At a few of the 1991 sites, laboratory sediment bioassays found a relationship between the exposure and uptake of certain metals by fish, as well as an ability of the sediments to induce benthic growth impairment (Bedard and Petro, 1992a). Work by Metcalfe-Smith *et al.*, (1995) also revealed that oils and greases were elevated in an area situated near an oil tank storage site. Even though the sediments received historical loadings of inorganic and organic contaminants, chemicals associated with the surficial sediment layer continue to pose a concern to local biota.

An integrated approach was used in the current 1997 investigation and involved the analysis of sediment contaminant levels for surficial and core samples, chemical body burdens in laboratory-exposed biota, spatial trends in toxicological effects associated with sediment physical and chemical properties and an evaluation on the health of the existing benthic population. The combined effort of both field and laboratory studies will provide a better understanding of the potential biological impact of sediment quality (Jaagumagi and Persaud, 1996). The project will also provide a current and more detailed account of the area to determine future remedial options (Richman, 1997).

This report provides the results and interpretation of whole-sediment toxicity tests conducted by OMOE, Standards Development Branch (SDB) following documented sediment toxicity test methods (Bedard *et al.*, 1992). The standardized laboratory tests were completed for seven sites using the mayfly nymph, *Hexagenia limbata* (21-day exposure, survival, growth and avoidance), the midge larvae, *Chironomus tentans* (10-day exposure, survival and growth) and the juvenile fathead minnow, *Pimephales promelas* (21-day exposure, survival and chemical bioaccumulation). The battery of sediment toxicity tests provides several endpoints using organisms representing different trophic levels in order to measure differences in sediment quality. The laboratory toxicity tests are a cost-effective technique for determining whether sediment-associated contaminants are harmful to benthic organisms or are being released into

the water column. In conjunction with appropriate negative and reference sediments, spatial differences in sediment quality, the relative availability of contaminants and their potential impacts can be ascertained. Sediment contaminant concentrations were based on samples prepared for laboratory toxicity testing. The sediment was analysed for particle size, nutrients, metals, total polychlorinated biphenyls (PCBs), total polycyclic aromatic hydrocarbons (PAHs), total petroleum hydrocarbons (TPHs), organochlorine pesticides and chlorinated benzenes. Surviving fathead minnows were submitted for whole-body tissue analysis of seven trace metals.

2.0 MATERIALS AND METHODS

2.1 Sample Collection and Site Description

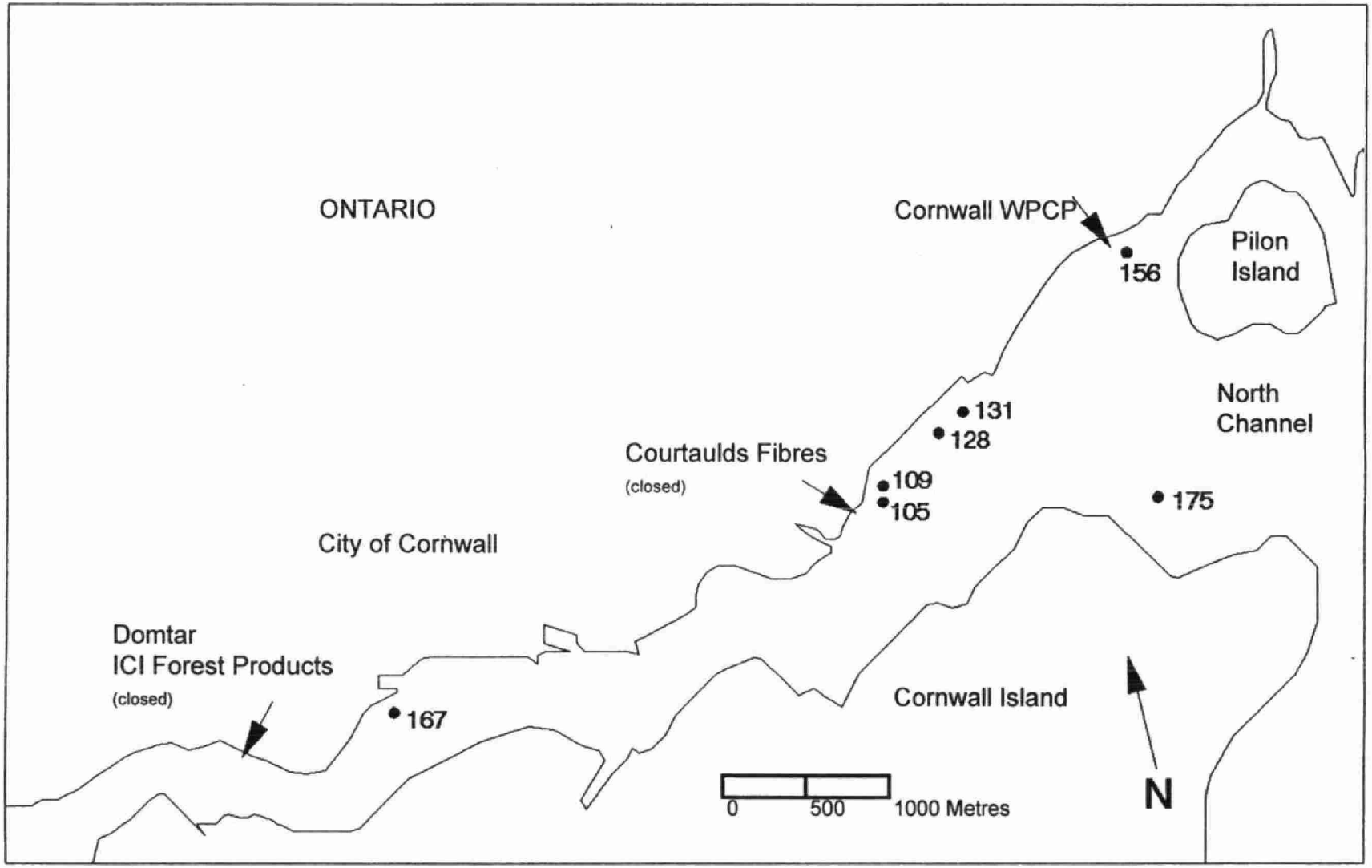
In late-October 1997, surficial sediment was collected at seven locations in the St. Lawrence River near Cornwall, Ontario (Figure 1; Table 1). The sampling locations were designated by OMOE, EMRB and Environment Canada. Site selection was based on the outcome of an earlier sediment survey conducted in 1994 (Richman, 1996) and covered areas of soft-textured sediments as identified using RoxAnn, a seabed classification system (Rukavina, 1996;1998). The seven sites designated for sediment toxicity testing was a subset of 24 stations which were chosen for an assessment of sediment chemistry (core and grab samples) and field benthic community analysis (Richman and Biberhofer, 1998; *in prep.*). The study area have previously been shown to have elevated Hg sediment concentrations (Richman 1994;1996), detectable levels of Hg in indigenous benthic species (Richman, 1994) and the potential for metal uptake and sublethal effects in sediment toxicity tests using fish and aquatic insects (Bedard and Petro, 1992a). In addition, high loadings of oils and greases have been measured in some sediment samples (Richman, 1996).

TABLE 1. 1997 St. Lawrence River Sediment Bioassay Station Locations

Station Number	Station Location
CS - 105	Northing: 4984819.6 Easting: 523938.0; Depth: 5.5 m; Offshore: 51 m
CS - 109	Northing: 4984878.7 Easting: 523978.8; Depth: 7.5 m; Offshore: 37 m
CS - 128	Northing: 4985063.9 Easting: 524479.0; Depth: 12 m; Offshore: 107 m
CS - 131	Northing: 4985169.2 Easting: 524567.2; Depth: 10 m; Offshore: 52 m
CS - 156	Northing: 4985718.0 Easting: 525505.1; Depth: NR; Offshore: 117 m
CS - 167	Northing: 4984039.0 Easting: 521149.9; Depth: 7.5 m; Offshore: 54 m
CS - 175	Northing: 4984852.3 Easting: 525541.7; Depth: 14 m; Offshore: 275 m

NR- Not Recorded;
Water depths recorded to the nearest 0.5 m; Offshore distance accurate to within 2 m.
(H. Biberhofer, EC, Pers. Comm.).

Figure 1. 1997 St. Lawrence River Sediment Sampling Stations



Sampling was done using a mini-ponar grab sampler. At each station, approximately 10 L of composited surficial sediment (top 10 cm) was collected from several grabs. The composited sediment was placed into 20 L plastic buckets lined with food-grade polyethylene bags and transported to the Toronto, Ontario laboratory and stored at 4°C until required.

Test sediment was collected in the following nearshore locations within the north channel above Cornwall Island (listed in order from upstream to downstream relative to the former Courtaulds Fibres shore-based acid recovery drain): 115 metres downstream (stn CS-105); 180 metres downstream (stn CS-109); 710 and 835 metres downstream (stns CS-128 and CS-131); 1.9 kilometres downstream and just west of Pilon Island (stn CS-156).

Station CS-167 was situated immediately downstream of the combined discharge of the former Domtar and ICI Forest Products plants, next to the Lamoureaux Park boat launch and about 2 kilometres upstream of Courtaulds Fibres.

Station CS-175 was collected off the northeastern point of Cornwall Island along the south shore of the north channel. Although the site is not considered to be representative of a 'pristine' area and strictly speaking cannot be used as a true 'reference control' sediment for the study, it was collected from a less contaminated area. A review of the sediment chemistry data show low sediment concentrations for those contaminants of concern, particularly Hg, Pb and Zn. The south shore site is also suspected to be less influenced by local sources relative to sites on the north shore due to current and flow patterns. The concentration of Hg was consistent with upstream reference sites from previous surveys in 1979, 1985 and 1991 (Kauss *et al.*, 1988; Anderson, 1990; Richman, 1994). For these reasons, stn CS-175 will be considered as a 'reference' site for comparison purposes rather than as a 'reference control'. Any physical similarities between the reference and test sites should also aid in data interpretation.

Sediment collected from Honey Harbour, Georgian Bay, Ontario served as the 'negative control'. The negative control sediment is a relatively uncontaminated sediment that provides a measure of test acceptability. The control is also a basis for comparing the biological responses from the test sediment (ASTM, 1997a).

2.2 Analytical Methods

Chemical analysis of sediment and biota samples was conducted by the OMOE, Laboratory Services Branch, located in Toronto. Test methods are described in the *OMOE Handbook of Analytical Methods for Environmental Samples* (OMOE, 1983). Quality assurance procedures included method blanks, spikes, duplicates and standard reference materials.

Sediment Nutrients and Particle Size Characterization

Homogenized bulk sediment (<2 mm fraction) was measured for total phosphorus (TP), total Kjeldahl nitrogen (TKN) and percent weight loss on ignition (LOI) which measured approximate organic content. Sediment total organic carbon (TOC) was determined with a LECO carbon analyzer using a dry combustion technique which oxidized carbon to CO_2. Particle size was measured on 50 g, air-dried samples using a Microtrac particle size analyzer for the size range 1.0 mm to 0.1 µm. This gives data on percent sand (2mm -62 µm), percent silt (62- 3.7 µm) and

percent clay (3.7 - 0.1 μm) size classes. Detailed test methodology is described in OMOEE (1995a; 1995b).

Trace Metals in Sediment

Prepared sediment samples were digested using a concentrated aqua-regia acid mixture (1 part HNO_3 to 3 parts HCl). The dissolved trace metals including As, Cd, Cr, Cu, Fe, Pb, Mn, Ni and Zn in the digestates were detected by inductively coupled argon plasma atomic emission spectroscopy (ICP-AES), and Hg by flow injection vapour generated flameless atomic absorption spectroscopy (AAS). Detailed test methodology is described in OMOEE (1994a).

Organic Chemicals in Sediment

Moist sediment samples were solvent-extracted with acetone and dichloromethane. The extract was transferred to a rotary evaporator, concentrated and fractionated on a Florisil column. Different solvent combinations were used to elute the extracts into three groups: fraction A1 contained total PCBs, five Aroclor groups, hexachlorobenzene, heptachlor, aldrin, octachlorostyrene, pp-DDE and mirex; fraction A2 contained a- & b-BHC, a- & b-chlordane, op-DDT, pp-DDD, pp-DDT; and fraction A3 included heptachlor epoxide, oxychlordane, dieldrin, endosulfan I & II, endosulfan sulphate, endrin and methoxyclor. Analytes were identified and quantified using capillary gas chromatography equipped with a Ni^{63} electron capture detector (GLC-ECD). Detailed test methodology is described in OMOEE (1994b).

Trace Metals in Biota

Pooled whole fish samples (~2.5 g) were thawed, homogenized and acid digested using a concentrated mixture of nitric acid (HNO_3) and perchloric acid ($HClO_4$) on triplicate samples per station. The digestate was concentrated by solvent evaporation on a heat block. The dissolved trace metals including Al, Cd, Cu, Pb, Mn, Ni and Zn in the digestates were detected by flow injection vapour generated flameless atomic absorption spectroscopy (AAS). Detailed test methodology is described in OMOEE (1996).

2.3 Laboratory Biological Testing Methods

Basic Experimental Design

Sediment biological tests were conducted according to OMOE standardized procedures (Bedard *et al.*, 1992) and are briefly described below. The bioassays were static, single-species tests using whole-sediment. The experimental unit was a 1.8 L test chamber containing prepared sediment and dechlorinated municipal tap water (1:4, v:v). The chambers were randomly placed into a holding tank at ambient room temperature and maintained under a 16:8 hour, light:dark photoperiod and continuous aeration.

Moist field-collected bottom sediment was pressed through a 2-mm stainless-steel sieve to remove existing large biota and debris prior to use. Sieving was completed from October 27 -

30, 1997. Subsamples of this homogenized sediment were submitted for chemical and physical characterization according to standard OMOE procedures (OMOE, 1989). The sieved sediment was homogenized with a spatula and stored in 4 L acid-rinsed glass jars until required. Three hundred and twenty-five millilitre aliquots of homogenized sediment were placed into the test chamber and overlaid with the test water. After settling overnight, the chambers were aerated continuously until the end of the test. A clean, negative control sediment was collected from Honey Harbour, Georgian Bay. Negative control mortality must not exceed 15% for mayflies and fathead minnows and 25% for chironomids or the test is declared invalid.

Water in the exposure chambers was regularly monitored for pH, conductivity, total ammonia, un-ionized ammonia and dissolved oxygen. Dead organisms were removed and the numbers recorded daily. Any signs of abnormal behaviour of the test organisms or changes in appearance of the test chambers were noted. Water loss due to evaporation was replenished as needed.

Hexagenia limbata Lethality and Growth Assay

The toxicity test used four month old laboratory-reared mayfly nymphs with an average wet weight of 4.63 mg ± 0.32 (s.e.) (n=44). The nymphs were raised from eggs collected by Dr. J. Ciborowski at the University of Windsor, Windsor, Ontario. Mayflies were reared according to OMOE procedures (Bedard *et al.*, 1992) and methods described in the literature (Friesen, 1981).

The rearing procedure involved the transfer of 600 newly-hatched nymphs to a 6.5 L aquarium which contained 2 cm of autoclaved sediment and 5.6 L dechlorinated tap water. Animals were maintained at ambient room temperature, 16:8 hour, light:dark photoperiod, continuous aeration and fed a vegetable diet.

Test organisms were retrieved from the rearing aquaria by sieving small portions of sediment in a 500-µm mesh brass sieve. The nymphs were washed into an enamel tray which held a fine mesh sieve and aerated, dechlorinated water. A Pasteur pipette (5-mm opening) was used to transfer the mayflies into 100 mL beakers of water and the contents were gently poured into the test chambers. Each test involved adding ten nymphs to each of the four replicate test chambers for a period of 21 days. Animals were not fed during the length of the test.

At the end of the test, the contents of each test chamber were emptied and rinsed in a sieve bucket. Surviving animals were counted and transferred to 150 mL beakers holding 100 mL dechlorinated water. The nymphs were immobilized with Alka-Seltzer®, blotted dry and individuals weighed to the nearest 0.01 mg.

Chironomus tentans Lethality and Growth Assay

Each toxicity test used 10-12 day old, cultured chironomid larvae weighing an average wet weight of less than 1 mg. The OMOE maintains continuous cultures of *C. tentans* larvae from egg to adult, following standard methods (Bedard *et al.*, 1992, Mosher *et al.*, 1982, Townsend *et al.*, 1981). Egg masses were originally supplied by Dr. J. Giesy at Michigan State University, Lansing, Michigan and have been cultured for several generations in our laboratory.

Initially, the midges were reared in enamel trays for 10 to 12 days and then maintained in a 21 L aquarium containing 1.6 L of silica sand. The cultures were held at ambient room temperature with continuous aeration and under a 16:8 hour, light:dark photoperiod. The larvae were provided a vegetable diet *ad libitum*.

Second and third instar larvae were directly transferred from the enamel rearing trays into the test chamber using the 5-mm opening of a Pasteur pipette. A total of 15 animals were added per chamber to each of the four replicates. Animals were fed daily 30 mg of a Cerophyll®:Tetra Conditioning Vegetable® (3:2, w:w) diet.

After 10 days, the contents of the test chambers were emptied and washed in a sieve bucket. Surviving animals were sorted, removed and placed into 150 mL beakers holding 100 mL dechlorinated water and 15 mL silica sand. The larvae were counted, blotted dry and individuals weighed to the nearest 0.01 mg.

Pimephales promelas Lethality and Bioaccumulation Assay

The tests used cultured, juvenile fathead minnows with an average wet weight of 437 mg ± 44 (s.e.) (n=16). The minnows were cultured at the OMOE laboratory following techniques which for the most part are US EPA procedures (USEPA, 1987) with minor revisions (Bedard *et al.*, 1992).

Cultures were maintained at 20°C in a flow-through dechlorinated water system and under a 16:8 hour, light:dark photoperiod. Breeders were kept in 60 L glass aquaria and eggs were laid on spawning tiles. The tiles were incubated in a 25°C water-bath and the developing larvae were transferred to 400 L fibreglass holding tanks. Larval fish were fed 48-hour old live brine shrimp while juveniles and breeders were provided frozen brine shrimp. Each size class was fed *ad libitum*.

Each test chamber received 10 juvenile minnows for each of the three replicates. The minnows were sorted into 250 mL glass beakers in groups of five. The contents of the beakers were emptied into a small net and the minnows released into the test chamber.

The minnows were exposed for 21 days and fed a NutraFin Staple® diet daily in an amount equivalent to 1% of the average starting wet weight. After 21 days the surviving fathead minnows were pooled from each replicate, counted, immobilized with Alka-Seltzer®, rinsed thoroughly with distilled water and placed into 30 mL glass vials and frozen pending chemical analysis.

Reference Toxicant Testing

A water-only reference toxicity ($CuSO_4$) test was conducted with *H. limbata* and *C. tentans* for 48-hours and LC50s were calculated. The static tests consisted of four test concentrations and a control. The nominal copper concentrations were 0.05, 0.25, 0.5, 1.0 and 3.0 mg/L. Ten mayfly nymphs or midge larvae were placed into each of four replicate 250 mL beakers. To help reduce stress, five glass tubes were placed into the mayfly test beakers and a fine layer of silica sand was added to the midge test containers. Water quality parameters were recorded at 0 and

48 hours. The mayfly test used four month old laboratory-reared mayfly nymphs with an average wet weight of 3.9 mg ± 0.4 (s.e.). The midge larvae were 10-12 day post-hatch with an average wet weight < 1 mg in each set of tests.

Bioassay Schedule for St. Lawrence River 1997 Sediment Samples

Test Organism	Species	Starting Date ('97/'98)	Completion Date ('97/'98)	Test Duration
Mayfly	*Hexagenia limbata*	November 5	November 26	21 days
Chironomid	*Chironomus tentans*	November 7	November 17	10 days
Minnow	*Pimephales promelas*	January 29	February 19	21 days

2.4 Statistical Methods

Statistical analyses were performed using the SAS® software package (SAS, 1985). Comparisons were made among the test and control sediments using One-Way Analysis of Variance (ANOVA) and Tukey's studentized range test (HSD) and planned comparisons (Steel and Torrie, 1960). Dunnett's one-tailed *t*-test was used solely to compare mortality between the control and test sediments and the associated minimum significant difference (MSD) was described as a measure of test sensitivity. Analysis was made on arc-sine transformed mortality data. Homogeneity of variance across groups was tested using Bartlett's test. Coefficients of variation (C.V. %) were calculated for each endpoint as a measure of test precision. Spearman rank correlation analysis was used to investigate the correlation among the different biological endpoints for each species and sediment characteristics. LC50's (including the associated 95% confidence limits) were calculated using software developed by Stephan (1977) and were estimated by probit analysis. Differences in whole-body tissue residues for metals among sites was determined using Tukey's studentized range test and statistical analysis was carried out on log-transformed data. Correlation analysis was used to measure the strength of the relationship between tissue and sediment chemical concentrations. All contaminant tissue residues were converted to a dry weight basis using a dry weight ratio of 0.15.

3.0 RESULTS

3.1 Water Quality Test Parameters

Conductivity, pH, total ammonia, un-ionized ammonia and dissolved oxygen parameters were periodically measured on the overlying water for each test species and summarized in Table 2. Values are reported as mean ± standard deviation.

TABLE 2. Mean (± s.d.) water quality characteristics in 1997 sediment bioassays.

Station	pH	D.O. mg/L	Conductivity umho/cm	Total Ammonia mg/L	Un-ionized Ammonia mg/L
Test Organism: Mayfly (Hexagenia limbata)[a]		Test Temperature: 20.0°C (0.6)			
Control	8.04 (.06)	8.6 (0.2)	317 (9)	<0.10 (0.0)	< 0.003
Stn CS-175	8.26 (.09)	8.5 (0.4)	455 (85)	0.22 (0.24)	0.012
Stn CS-105	8.28 (.08)	8.4 (0.3)	481 (100)	0.49 (0.73)	0.038
Stn CS-109	8.28 (.05)	8.4 (0.4)	516 (108)	0.41 (0.58)	0.031
Stn CS-128	8.31 (.12)	8.4 (0.3)	473 (91)	0.45 (0.63)	0.022
Stn CS-131	8.27 (.20)	8.4 (0.4)	479 (83)	0.75 (0.85)	0.040
Stn CS-156	8.28 (.02)	8.5 (0.4)	451 (82)	0.25 (0.30)	0.020
Stn CS-167	8.18 (.06)	8.5 (0.4)	393 (44)	0.45 (0.70)	0.019
Test Organism: Midge (Chironomus tentans)[b]		Test Temperature: 19.7°C (0.6)			
Control	7.93 (.22)	8.2 (0.6)	331 (11)	0.20 (0.17)	0.005
Stn CS-175	8.00 (.12)	8.0 (0.7)	418 (62)	0.51 (0.45)	0.015
Stn CS-105	8.22 (.03)	8.3 (0.6)	429 (48)	1.10 (0.87)	0.060
Stn CS-109	8.11 (.26)	8.0 (0.7)	452 (63)	1.16 (1.00)	0.037
Stn CS-128	8.13 (.20)	8.1 (0.7)	431 (55)	1.23 (1.05)	0.047
Stn CS-131	8.21 (.05)	8.4 (0.5)	435 (48)	1.66 (1.46)	0.100
Stn CS-156	8.01 (.36)	8.2 (0.6)	399 (52)	0.83 (0.75)	0.018
Stn CS-167	8.07 (.01)	8.6 (0.5)	370 (25)	0.80 (0.75)	0.029
Test Organism: Minnow (Pimephales promelas)[a]		Test Temperature: 20.7°C (0.3)			
Control	7.20 (.16)	6.2 (1.7)	351 (29)	0.66 (0.56)	0.006
Stn CS-175	7.59 (.08)	7.5 (0.1)	520 (103)	0.94 (1.12)	0.012
Stn CS-105	7.61 (.15)	7.3 (0.4)	537 (116)	3.80 (1.93)	0.055
Stn CS-109	7.55 (.14)	7.2 (0.4)	566 (112)	4.90 (2.40)	0.110
Stn CS-128	7.69 (.20)	7.0 (0.6)	511 (82)	2.10 (2.08)	0.068
Stn CS-131	7.69 (.14)	7.5 (0.1)	531 (95)	3.00 (2.57)	0.074
Stn CS-156	7.59 (.10)	7.4 (0.3)	486 (84)	1.15 (1.66)	0.016
Stn CS-167	7.56 (.15)	7.2 (0.8)	428 (50)	2.44 (2.11)	0.033

a Sample size N=4; b Sample size N=3;

Underlining indicates un-ionized ammonia concentrations that exceed the PWQO of 0.02 mg/L

The water quality measurements were similar among the test sites, regardless of test species or test duration. The average pH recorded among the reference and six test sediments for the mayfly, midge and minnow tests were 8.2, 8.1 and 7.6, respectively. Conductivity averaged 464 µmho/cm, 419 µmho/cm and 511 µmho/cm for the three species. Dissolved oxygen within the test jars remained above the minimum acceptable level (>4 mg/l) throughout the test (OMOEE, 1994c). Test temperature was at 20°C for each bioassay.

The amount of total ammonia (NH_4), along with the converted un-ionized ammonia (NH_3) based on temperature and pH, were also recorded in Table 2. The lowest un-ionized ammonia concentration occurred for the negative control exposure in each toxicity test (0.003 to 0.006 mgNH_3/L), followed by the reference exposures (0.012 to 0.015 mgNH_3/L). Most of the test sediments had average un-ionized concentrations that exceeded the PWQO of 0.02 mgNH_3/L in each test. Concentrations ranged from 0.03 mgNH_3/L in the mayfly test to 0.06 mgNH_3/L in the minnow test. The midge test had an intermediate concentration of 0.05 mgNH_3/L. The residual amounts of ammonia present in the overlying water was most likely derived from the test sediment rather than directly from the biota, given the consistency observed among the three test species. All the test sediments, with the exception of stn CS-128, had twice the amount of TKN relative to the reference sediment, which may help to explain the differences in ammonia concentrations between reference and test sites. The un-ionized ammonia concentrations are well below cited acute and chronic concentrations for fathead minnows (Thurston *et al.*, 1983; 1986) and midge larvae (Schubauer-Berigan *et al.*, 1995; Whiteman *et al.*, 1996), recorded for, or extrapolated using the actual test temperature and pH.

3.2 Sediment Characterization

The following sections summarize the sediment physical and chemical parameters to aid in the interpretation of the biological toxicity results. Chemical analysis is based on the sediment prepared for toxicity testing and results may differ from those reported for any field samples collected concurrently. Any dissimilarities are likely due to *in-situ* chemical heterogeneity and/or sampling depth and sample handling (ASTM, 1997b).

Physical and Nutrient Properties

Sediments were characterized for % sand (2mm-62µm), % silt (62-3.7µm), % clay (3.7-0.1µm), % loss on ignition (LOI), total organic carbon (TOC), total phosphorus (TP) and total Kjeldahl nitrogen (TKN) (Table 3).

Most of the test sediments, along with the reference and negative control, represented the same substrate type e.g. loam (Millar *et al.*, 1965). Of these sediments, the sand fraction consisted primarily of finer-grade size particles of <125 µm. Only stn CS-105 had a higher proportion of coarser-sized sand particles (>352 µm). The sediments were very black in colour, soft-textured and often had present small quantities of wood chips and fibres. Station CS-109 sediment had the highest percent silt and classified as a silty, clay loam.

Three of the test sediments (stns CS-105, -131, -156) contained intact fingernail clams and broken shell fragments. Similarly, the reference sediment (stn CS-175) also indicated the presence of clams along with endemic benthos including midges and oligochaetes. During initial

TABLE 3. Sediment physical and nutrient characteristics in control and St. Lawrence River 1997 sediment used in sediment bioassays.

Station	*% Sand* (2mm-62um)	*% Silt* (62-3.7um)	*% Clay* (3.7-0.1um)	*% LOI*	*TOC* mg/g	*TP* mg/g	*TKN* mg/g
Honey Harbour Control	33.0	44.0	22.3	8.6	40	1.0	3.1
Station CS - 175 Reference	41.0	36.9	22.2	7.3	36	0.5	1.1
St. Lawrence River							
Station CS - 105	53.0	31.1	16.1	6.3	30	0.9	2.5
Station CS - 109	12.0	59.5	29.4	12.0	41	1.1	3.5
Station CS - 128	50.0	35.4	14.2	14.0	24	0.8	0.7
Station CS - 131	31.0	44.8	23.8	7.3	36	1.0	1.9
Station CS - 156	51.0	33.2	15.1	5.4	24	0.9	2.4
Station CS - 167	45.0	40.2	14.7	7.7	42	0.9	2.8
PSQG SEL Conc (mg/g dry weight)					100	2.0	4.8

sieving, foreign matter was noted at stn CS-109 in the form of synthetic fibres.

In terms of nutrient content, the test sediments had varying amounts of organic carbon ranging from 24 mg/g to 42 mg/g TOC and organic matter of 5.4 to 14.0% LOI. No exceedences of TP or TKN above the SEL were found. Sediment particle size did not significantly correlate with differences in sediment TOC ($r=0.65$; $p=.10$; $n=7$), however, the presence of woody detrital material may in part have contributed to the final TOC measurements. The moderate amount of TOC in the reference sediment (36 mg/g) should provide a reasonable measure of biological response due to maximum nutrient enrichment relative to the test sites.

Trace Metal Sediment Concentrations

Bulk sediment samples were analysed for 11 trace metals (Table 4). The sediment metal concentrations were compared to Severe Effect Level (SEL) and Lowest Effect Level (LEL) concentrations as outlined in the Provincial Sediment Quality Guidelines (PSQGs) (Persaud *et al.*, 1992). The SEL is defined as that chemical concentration in the sediment that could be detrimental to the majority of the macrobenthos and the LEL is the sediment contaminant concentration which can be tolerated by most benthic species.

Mercury sediment concentrations most frequently surpassed the PSQG-SEL concentration of 2 µg/g. Sediments collected ~700 metres downstream of the former Courtaulds Fibres plant had the highest Hg concentration, Stn CS-128; 6.1 µg/g and Stn CS-131; 5.4 µg/g. One of the stations adjacent to the Courtaulds site also had Hg sediment concentration above the SEL level. The remaining three test sites were above LEL concentrations (Hg: 0.7 to 1.5 µg/g). The observed Hg sediment concentrations fell within those previously reported for the same area (Dreier *et al.*, 1997). Analysis of core samples have confirmed local nearshore sediments have been historically impacted from dischargers including Courtaulds Fibres (Richman, 1996). A maximum Hg sediment concentration of 13.8 µg/g was found in 1994, measured in the top 10 cm section of a core sample with other values typically ranging from 2.0 µg/g to 11.9 µg/g (Richman, 1996). Richman (1994) reported a maximum Hg sediment concentration of 3.1 µg/g for a surficial, top 3 cm sample in 1991 and an average concentration of 1.7 µg/g Hg at six locations corresponding to the same study area as those described herein.

The only other metal exceedence occurred for Zn at Stn CS-109 with a concentration of 1,200 µg/g. Cd, Cr, Cu, Ni and Pb were commonly measured above LEL sediment concentrations and As, Fe and Mn below LEL concentrations and approximate those levels found in the reference sediment.

Organic Chemical Sediment Concentrations

Concentrations of 19 organochlorine pesticides and 8 chlorinated organic compounds in all sediment samples were below the respective detection limits (Table 5). Those compounds measured at trace concentrations for some sites included, pp-DDE, 1,3,5-trichlorobenzene, 1,2,3,5-tetrachlorobenzene and hexachloroethane (Table 6). Trace amounts of hexachlorobenzene (2 - 9 ng/g) were found at all the test sites. Measurable concentrations of hexachlorobenzene of 11 ng/g and 1,2,4,5-tetrachlorobenzene (10 and 13 ng/g) were occasionally recorded.

TABLE 4. Bulk concentrations of trace metals in control and St. Lawrence River 1997 sediment (µg/g dry weight) used in sediment bioassays.

Station	*Al %*	*As*	*Cd*	*Cr*	*Cu*	*Fe %*	*Hg*	*Mn*	*Ni*	*Pb*	*Zn*
Honey Harbour Control	2.2	5.0	1.4	44	24	3.7	0.09	980	35	54	140
Station CS - 175 Reference	1.6	3.3	1.5	40	41	2.3	0.1	400	28	30	140
St. Lawrence River											
Station CS - 105	1.2	4.0	1.5	41	55	1.8	1.5	270	25	70	750
Station CS - 109	1.3	4.8	1.7	55	85	2.0	3.6	290	32	170	1200
Station CS - 128	0.6	2.6	0.7 <T	18	26	1.2	6.1	260	13	21	100
Station CS - 131	1.0	4.3	1.3	34	51	1.6	5.4	280	23	49	280
Station CS - 156	0.8	3.0	0.8 <T	26	28	1.4	0.7	260	19	31	180
Station CS - 167	1.0	3.6	1.5	33	40	1.6	1.0	220	25	39	120
PSQG SEL Conc.	NA	33	10	110	110	4.0	2.0	1100	75	250	820
PSQG LEL Conc.	NA	6.0	0.6	26	16	2.0	0.2	460	16	31	120

<T - Trace Amount; Shading indicate sediment trace metal concentrations that exceed PSQG-SELs.

Underlining indicate sediment trace metal concentrations that exceed PSQG-LELs; NA - Not Available.

TABLE 5. Bulk sediment concentrations for chlorinated organics and pesticides in reference and St. Lawrence River 1997 sediment (ng/g, dry weight) used in bioassays.

All Stations	Heptachlor	1 <W
	Aldrin	1 <W
	Mirex	5 <W
	a-BHC	1 <W
	b-BHC	1 <W
	g-BHC	1 <W
	a-Chlordane	2 <W
	g-Chlordane	2 <W
	Oxychlordane	2 <W
	op-DDT	5 <W
	pp-DDD	5 <W
	pp-DDT	5 <W
	Methoxychlor	5 <W
	Heptachlor epoxide	1 <W
	Endosulphan I	2 <W
	Dieldrin	2 <W
	Endrin	4 <W
	Endosulphan II	4 <W
	Endosulphan sulphate	4 <W
	Hexachlorobutadiene	1 <W
	Octachlorostyrene	1 <W
	123-Trichlorobenzene	2 <W
	124-Trichlorobenzene	2 <W
	1234-Tetrachlorobenzene	1 <W
	Pentachlorobenzene	1 <W
	236-Trichlorotoluene	1 <W
	245-Trichlorotoluene	1 <W

<W - Not Detected.

TABLE 6. Concentrations of selected organic compounds in St. Lawrence River 1997 sediments (ng/g, dry weight) used in sediment bioassays.

Station	*HCB*	*pp-DDE*	*1,2,3,5 TeCB*	*1,2,4,5 TeCB*	*1,3,5- TCB*	*HCE*
Station CS - 175 Reference	1 <W	3 <T	1 <W	1 <W	2 <W	1 <W
St. Lawrence River						
Station CS - 105	3 <T	1 <W	1 <W	1 <W	2 <W	1 <W
Station CS - 109	3 <T	1 <W	1 <W	1 <W	2 <W	1 <W
Station CS - 128	4 <T	2 <T	1 <W	10	16 <T	2 <T
Station CS - 131	11	5 <T	2 <T	7 <T	12 <T	3 <T
Station CS - 156	2 <T	1 <W	1 <W	7 <T	2 <W	4 <T
Station CS - 167	9 <T	1 <W	2 <T	13	8 <T	3 <T

< W - Not Detected; <T - Trace Amount; HCB - Hexachlorobenzene; HCE - Hexachloroethane

Most of the test sediments had a characteristic petroleum-like odor, oily sheen and viscous blobs that stained laboratory equipment. The exact source of this material is unclear since it did not occur as PAH, TPH or PCBs at high concentrations (Table 7). Individual and total PAH sediment concentrations were fairly similar among sites. Total PAH sediment concentrations were near the PSQG-LEL of 4.0 µg/g. At least five of the individual PAH compounds were measured at trace amounts in each sample. The reference and negative control sediment had the lowest total PAH concentrations of 1.2 µg/g <T and 0.4 µg/g <T, respectively. No single PAH constituted more than 20% of the sum total of the 16 PAHs across the study area suggesting little or no PAH enrichment. Similarly, TPH concentrations were measured below the detection limit of 100 µg/g which is not surprising since it is considered a subset of total PAHs. Total PCBs ranged from 60 ng/g to 360 ng/g and exceeded the LEL of 70 ng/g at all test sites but were at least 1/70th lower than the SEL concentration (corrected for organic carbon).

3.3 Mayfly (*Hexagenia limbata*) 21-day Lethality and Growth Results

The biological data for the two endpoints, mortality and growth, are summarized in Table 8. No significant differences in mayfly mortality were found among the test, reference and negative control (ANOVA; $p<0.37$). Percent mortality ranged from 0% to 10% and all samples were ranked similarly using Tukey's multiple range test.

Mayfly body weights varied significantly among test and control sediments (ANOVA; $p<0.0001$). Final growth measurements fell into two distinct groups using planned comparisons as illustrated in Figure 2. The best nymphal growth occurred at four sites (stns CS-105, -156, -109, -167) and was on par with the level of growth observed for the reference sediment (Stn CS-175). The remaining test sediments resulted in mayfly weights which were comparable to the negative control but were lower than the reference sediment (stn CS-175) by 47% (stn CS-131) and 27% (stn CS-128), as well as against the other test sediments. Observations made throughout the bioassay did not indicate any abnormal organism avoidance behaviour.

The relatively low body weight of 6.2 mg found for the negative control animals is likely attributed to the longer sediment storage time (~12 months) versus the freshly collected test sediments (~ 2 weeks). The prolonged storage may have affected the nutritive value of the sediment, thereby altering feeding rates (Boese *et al.,* 1996). Mayfly growth depends on the quality and quantity of detrital material found in the sediment, since a supplemental diet was not provided during the test. Therefore, the reference sediment would be the better indicator of mayfly growth capability given similar storage conditions and high organic content.

3.4 Chironomid (*Chironomus tentans*) 10-day Lethality and Growth Results

Results for chironomid growth and lethality are reported in Table 8 and the sublethal data illustrated in Figure 2. Chironomid control percent mortality was 0% and 6% for the reference and negative control, respectively. These values are well below the maximum acceptable criterion of 25% mortality. Percent mortality for each of the test sediments of 1% to 8% mortality was ranked similarly with both controls, with the exception of stn CS-156. Station CS-156 percent mortality of 26% was just significantly higher than the reference (stn CS-175) ($p<0.05$) mortality but was not significantly different to the negative control (Dunnett's *t*-test).

TABLE 7. Bulk concentrations of polycyclic aromatic hydrocarbons (ng/g), total polychlorinated biphenyls (ng/g) and total petroleum hydrocarbons (µg/g) in St. Lawrence River 1997 sediment.
Concentrations reported as dry weight.

Parameter	Reference CS - 175	Cornwall CS - 105	Cornwall CS - 109	Cornwall CS - 128	Cornwall CS - 131	Cornwall CS - 156	Cornwall CS - 167
Acenaphthene	20 <W	120 <T	76 <T	38 <T	44 <T	68 <T	42 <T
Acenaphythylene	22 <T	110 <T	38 <T	20 <W	23 <T	44 <T	31 <T
Anthracene	20 <W	260	270	61 <T	96 <T	170 <T	140 <T
Benzo[a]anthracene	160 <T	610	940	160 <T	370	720	490
Benzo[b]fluoranthene	180 <T	1000	1600	180 <T	450	740	700
Benzo[k]fluoranthene	45 <T	310	440	70 <T	160 <T	150 <T	180 <T
Benzo[ghi]perylene	91 <T	580	780	86 <T	210	390	360
Benzo[a]pyrene	140 <T	710	1100	140 <T	360	610	530
Chrysene	100 <T	750	1000	140 <T	480	830	480
Dibenzo[ah]anthracene	40 <W	170 <T	150 <T	40 <W	60 <T	110 <T	74 <T
Fluoranthene	140 <T	1200	2100	320	630	850	820
Fluorene	20 <W	160 <T	100 <T	42 <T	60 <T	89 <T	62 <T
Indeno[123-cd]pyrene	120 <T	710	1000	100 <T	250 <T	410	420
Naphthalene	20 <W	120 <T	55 <T	20 <W	25 <T	36 <T	37 <T
Phenanthrene	56 <T	620	970	280	450	480	430
Pyrene	150 <T	1000	1600	340	690	990	730
Total PAHs	1204 <T	8430	12219	1957 <T	4358	6687	5526
Total PCBs	60	200	360	100	200	160	160
TPHs	100 <W	100 <W	100 <W	100 <W	100 <W	100 <W	100 <W

<W - Not Detected; <T - Trace Amount; Underlining indicate sediment PAH and PCB concentrations that exceed PSQG-LELs; PSQG's not available for acenapthene, acenaphthylene, benzo[b]flouranthene and naphthalene.

TABLE 8. Summary of biological results on mayfly, midge and minnow sediment bioassays for control and St. Lawrence River 1997 sediments.

Mean values (± standard deviation) where sample size n=4 replicates for mayfly and midge; n=3 for minnow tests.

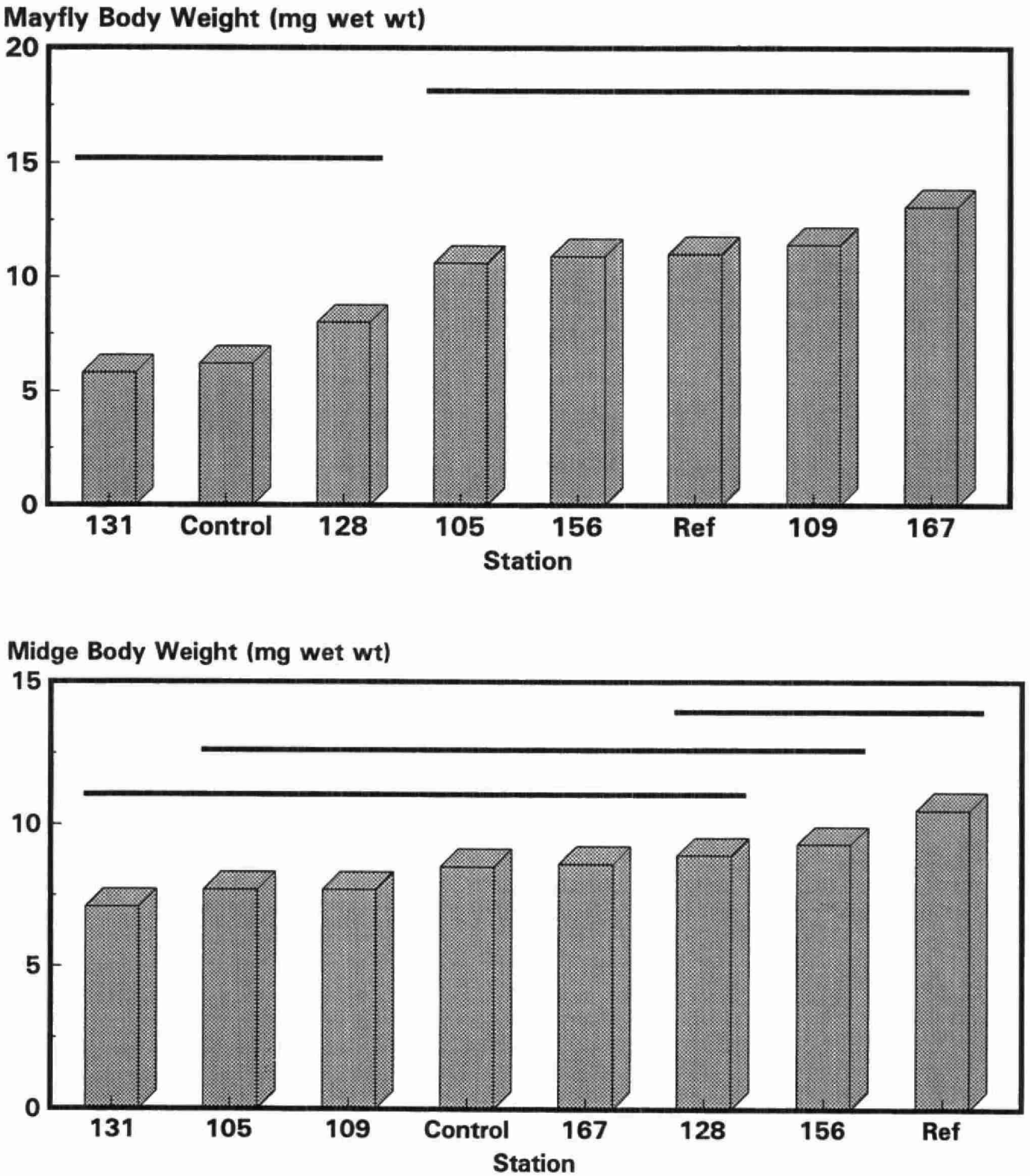
Test Organism	*Hexagenia limbata* (Mayfly)		*Chironomus tentans* (Midge)		*Pimephales promelas* (Fathead Minnow)
Station	*% Mortality*	*Ave. Individual Body Weight* (mg wet wt.)	*% Mortality*	*Ave. Individual Body Weight* (mg wet wt.)	*% Mortality*
Honey Harbour Control	A 0 (0)	B 6.21 (0.6)	AB 6.6 (5)	BC 8.49 (0.8)	A 0 (0)
Stn CS - 175 Reference	A 0 (0)	A 11.05 (1.1)	A 0 (0)	A 10.53 (1.3)	A 0 (0)
St. Lawrence R. Stn CS - 105	A 0 (0)	A 10.66 (1.1)	AB 1.6 (3)	BC 7.69 (0.7)	A 0 (0)
Stn CS - 109	A 2.5 (5)	A 11.39 (1.0)	AB 8.3 (10)	BC 7.72 (0.9)	A 0 (0)
Stn CS - 128	A 5.0 (10)	B 8.05 (0.8)	AB 6.6 (5)	ABC 8.92 (0.6)	A 0 (0)
Stn CS - 131	A 10.0 (14)	B 5.82 (0.2)	AB 1.6 (3)	C 7.18 (1.3)	A 0 (0)
Stn CS - 156	A 0 (0)	A 10.95 (0.4)	B * 26.6 (28)	AB 9.34 (2.4)	A 0 (0)
Stn CS - 167	A 2.5 (5)	A 13.17 (2.9)	AB 1.6 (3)	BC 8.59 (0.7)	A 0 (0)
% MSD	11.6	-	19.5	-	-
% C.V.	171.4	10.5	112.1	13.0	-
D.P.	2.0	6.8	3.5	2.9	-

* %Mortality value is significantly different than the reference control sediment (Dunnett's 1-tailed t-test;$p<0.05$).

A Means sharing a common letter within a column are not significantly different; Tukey's HSD test for % Mortality ($p<0.05$) and planned comparisons using LSMEANS for comparing Body Weight ($p<0.01$).

MSD - Minimum Significant Difference; C.V. - Coefficient of Variation; D.P. - Discriminatory Power.

Figure 2. Mayfly and Midge Growth for St. Lawrence River 1997 Sediments



Lines indicate significant groups.

Midge growth differed significantly, depending on the sediment involved in the comparison (ANOVA; $p<0.0001$). Midge body weight measured for each of the test sediments belonged to a group that overlapped with all other groupings, indicating no obvious trends in growth among sites. It appears the degree of growth achieved for the reference sediment is indicative of optimal conditions and resulted in the largest-sized animals of 10.5 mg. The negative control sediment promoted an average level of midge growth of 8.5 mg and either sediment can be treated as an adequate representation of midge growth potential. Among site comparisons indicated each of the test sediments promoted midge growth that ranged from average to above average.

3.5 Fathead Minnow (*Pimephales promelas*) 21-day Lethality Results

Juvenile fathead minnow percent mortality data is reported in Table 8. Minnow survival was 100% in the negative control, reference and all test sediments. A rough estimate of fish wet weight was made on pooled samples on Day-21. The final fresh weights for each batch of test animals did not differ more than 10% of the combined weights measured for the control and reference exposures. The equality in minnow weight across sites suggests the fish were feeding normally and the sediments were not contributing to underlying stress, assuming body weight is an indicator of fish condition.

3.6 Quality Assurance Data

An evaluation was made on the biological data in order to determine the repeatability of the test results (Table 8). Excellent test precision was apparent for two of the test endpoints according to the range in coefficient of variation. Mayfly growth and midge growth exhibited a high degree of test accuracy with % C.V. ranging from 10% to 13%. Between these endpoints, mayfly growth also had the highest discriminatory power of D.P.=7. This was evident by the distinction made between those sites with normal versus substandard growth using *Hexagenia*. Mayfly survival, midge survival, midge growth and minnow survival endpoints were less sensitive in determining differences among sites as reflected in the outcome of the bioassay results.

Each of the lethality responses was effective in detecting small differences in lethality among sites, as shown by the excellent minimum significant difference (MSD) values of 11% and 19%. No determination was possible for the minnow bioassay due to the lack of an effect. Other toxicity tests that followed OMOE standard test methods, had similar reported average MSDs of 12% (*H. limbata*; n=8) and 18% (*C. tentans*; n=9) (D. Bedard, OMOE, unpublished data). The MSD is a quantitative measure of test precision which describes the ability to detect a significant effect in the paired response between the control versus the test sample. The test design of each toxicity test was suitable in detecting small differences in lethality as being significant yet all the test sediments were found to be non-toxic, irrespective of the test species employed.

The 48-hour copper LC50 (95% C.I.) for the water-only reference toxicant exposures for *H. limbata* was 0.50 (0.31 - 0.69) mg/L. This value was within the acceptable 48-h LC50 (± 2 s.d.) range of 1.14 (1.32) mg/L, according to a previous series of reference toxicant tests. Similarly, for *C. tentans*, the LC50 was 1.45 (1.16 - 1.89) mg/L, as compared to an expected 48-h LC50 (± 2 s.d.) of 1.19 (0.91) mg/L. This indicates that the relative sensitivity of the test organisms fell within a normal response range.

3.7 Chemical Bioaccumulation in *Pimephales promelas*

The examination of chemical availability to aquatic organisms is valuable for assessing the potential for chemical transfer through the food web. The primary objective of this test design is to make general observations on whole organism tissue concentrations as they relate to bulk chemical concentrations in the sediment and differences in chemical uptake among sites. Surviving fathead minnows were submitted for the analysis of trace metals. All values are based on whole-body tissue residues. Values are provided as wet weight and converted using a dry to wet ratio of 0.15 in the calculation of biota-sediment accumulation factors or BSAFs.

The sources of inorganic accumulation to forage fish include direct contact with the sediment and uptake from the overlying water. Factors that control chemical accumulation by forage fish include those that affect chemical speciation, adsorption and desorption such as sediment organic content, redox potential, pH, Fe and Mn oxide and particle size distribution (Luoma, 1983). Another important factor to consider in describing the chemical bioavailability of certain divalent trace metals is acid-volatile sulfides (AVS) (Ankley, 1996). Biotic factors affecting uptake include metabolism and metal specific toxicokinetics (Campbell *et al.*, 1988).

Table 9 reports the Cd, Cu, Hg, Mn, Ni, Pb and Zn tissue concentrations (wet weight) and associated standard deviations measured in surviving juvenile fathead minnows. Whole-organism chemical concentrations are based on triplicate samples. The fathead minnow bioassay was delayed due to animal availability. Testing began approximately three months after sample collection. To help minimize any possible changes in sediment integrity during storage, every attempt should be made to begin testing soon after collection and up to a maximum permissible time frame of six weeks (EC, 1994; Bedard *et al.*, 1992). Given the uncertainties of sediment integrity and chemistry alterations due to prolonged storage (ASTM, 1997b), another set of reference and test sediments were submitted for metal and TOC analyses (Table 10).

Of the 49 individual measurements taken for the seven trace metals, 85% remained within 10% of their original value based on samples that were stored for two weeks (see Table 4). This indicates a high degree of stability for total metal concentrations but not necessarily with respect to metal speciation. Both losses and gains of less than 10% were reported among the metals and is likely a reflection of sediment heterogeneity since it applied equally to both reference and test sediments. The single largest difference was most frequently noted at those sites where Cd was measured in trace amounts, as well as for Hg at four test sites. Total Hg sediment concentrations increased by 16% for stn CS-167 and by 37% for stn CS-131, as compared to the freshly stored samples. Similarly, decreases in Hg occurred at stns CS-128 of 16% and 28% at stn CS-156. The reason for the large spread in percent change in sediment Hg could be due to chemical heterogeneity but it was also noted that differences in Hg sediment concentrations coincided with changes in TOC values. There was an inverse relationship between TOC and Hg. A decrease in TOC tended to result in higher total Hg sediment concentration. Each station when placed in order from high to low Hg sediment concentrations were relatively unchanged between the two storage dates. A switch in rankings occurred for the two most contaminated sites, stns CS-128 and CS-131, between the two week and three month storage periods. No alterations in LEL and SEL assignments were found.

There were significant differences in fish tissue concentrations among the control and test sediments for each of the trace metals (One-way ANOVA; $p=0.04$ to $p=0.00001$). Irrespective of any specific differences, for the most part, tissue concentrations attained in the test exposures

TABLE 9. Mean metal concentrations in fathead minnows exposed to control and St. Lawrence River 1997 sediments in the laboratory and associated biota-sediment accumulation factors (BSAFs).

Tissue concentrations reported as µg/g wet weight; Mean values ± standard deviation; Sample size n=3.

Station	Cd	Cu	Hg	Mn	Ni	Pb	Zn
Pre-exposure	0.04 (.01)	0.89 (.03)	0.09 (.01)	1.7 (.07)	0.02 <W (.00)	0.02 <W (.00)	39.5 (0.7)
Georgian Bay Control	A 0.10 (.04)	A 0.96 (.11)	A 0.08 (.01)	C 7.3 (.94)	AB 0.17 (.13)	AB 0.15 (.18)	AB 43.3 (2.5)
Reference Station CS-175	AB 0.03 (.03)	BC 1.30 (.10)	A 0.06 (.01)	B 4.8 (.30)	BC 0.47 (.08)	AB 0.15 (.11)	A 42.3 (1.5)
BSAF	0.14	0.21	4.30	0.08	0.10	0.03	2.14
Station CS-105	AB 0.06 (.02)	BC 1.20 (.05)	A 0.06 (.01)	A 2.7 (.20)	A 0.12 (.13)	B 0.55 (.24)	AB 46.0 (3.0)
BSAF	0.29	0.15	0.27	0.06	0.03	0.05	0.40
Station CS-109	B 0.01 (.01)	D 1.66 (.05)	A 0.08 (.02)	A 3.3 (.10)	ABC 0.36 (.10)	C 1.40 (.20)	B 50.0 (2.6)
BSAF	0.03	0.12	0.13	0.07	0.08	0.05	0.27
Station CS-128	AB 0.04 (.03)	A 0.97 (.03)	B 0.17 (.03)	A 2.8 (.11)	AB 0.12 (.11)	A 0.07 (.08)	A 41.0 (1.7)
BSAF	0.36	0.26	0.21	0.07	0.06	0.02	2.45
Station CS-131	AB 0.02 (.03)	BC 1.20 (.11)	AB 0.12 (.04)	A 2.8 (.30)	A 0.06 (.05)	AB 0.23 (.22)	A 42.3 (2.0)
BSAF	0.12	0.15	0.09	0.06	0.02	0.03	0.99
Station CS-156	AB 0.05 (.02)	AB 1.13 (.11)	A 0.08 (.01)	A 3.1 (.49)	AB 0.14 (.13)	AB 0.42 (.15)	AB 46.3 (1.5)
BSAF	0.31	0.26	1.30	0.08	0.05	0.08	1.69
Station CS-167	AB 0.09 (.03)	CD 1.46 (.05)	A 0.08 (.00)	A 3.0 (.28)	C 0.55 (.17)	AB 0.51 (.17)	AB 44.6 (3.0)
BSAF	0.39	0.24	0.43	0.09	0.15	0.08	2.45

A Means sharing a common letter within a column are not significantly different using Tukey's HSD test ($p<0.05$).

TABLE 10. Bulk concentrations of trace metals and TOC in reference and St. Lawrence River sediment that was stored and used in the calculation of biota-sediment accumulation factors.

Station	TOC mg/g	*Cd* µg/g	*Cu* µg/g	*Hg* µg/g	*Mn* µg/g	*Ni* µg/g	*Pb* µg/g	*Zn* µg/g
Station CS - 175 Reference	37	1.5	39	0.1	370	28	32	130
St. Lawrence River								
Station CS - 105	34	1.4	55	1.5	270	25	71	750
Station CS - 109	37	2.0	88	3.8	280	29	180	1200
Station CS - 128	27	0.8	24	5.1	260	13	23	110
Station CS - 131	36	1.4	53	8.6	280	23	54	280
Station CS - 156	29	1.1	28	0.4	250	18	32	180
Station CS - 167	41	1.5	40	1.2	210	24	39	120
PSQG SEL Conc.	100	10	110	2.0	1100	75	250	820
PSQG LEL Conc.	10	0.6	16	0.2	460	16	31	120

<T - Trace Amount; Shading indicate sediment trace metal concentrations that exceed PSQG-SELs.
Underlining indicate sediment trace metal concentrations that exceed PSQG-LELs; NA - Not Available.

were equivalent to background concentrations represented as pre-exposure conditions or local reference conditions. For instance, Ni and Cd tissue concentrations in test animals matched those attained in the reference animals and were indicative of baseline values. Manganese consistently attained tissue concentrations in the test sediments that were less than the reference animals. The above three metals failed to show any significant relationship in metal tissue concentration relative to the corresponding metal sediment concentration (Table 11).

Among the metals, Pb tissue concentrations exhibited the strongest dependency to the concentration measured in the sediment ($r=0.86$; $p=0.03$). The high degree of variability in Pb tissue concentrations among sites, by as much as 20-fold, reflected the fairly wide range in sediment concentrations which differed by as much as 87% between the highest and the lowest concentrations. Both of these factors suggest that increasing metal sediment concentrations associated with any sediment contained within the gut of the organism could have lead to a correspondingly increasing Pb concentration at the whole-organism level.

In contrast to Pb, Cu tissue concentrations remained fairly steady (Range: 0.9 to 1.6 µg/g, wet wt) despite the variability observed in sediment Cu concentrations. Several studies have shown the capacity of Cu to be partially regulated in aquatic organisms such as those described by Borgmann *et al.*, (1993) and Borgmann and Norwood (1997) using the freshwater amphipod, *Hyalella azteca*.

Metal regulation was most clearly illustrated by the pattern in Zn uptake by fathead minnows. Tissue Zn concentrations varied by only 1.2-fold among sites, while sediment Zn concentrations varied by at least 10-fold. Zinc is considered an essential micronutrient and can be homoeostatically regulated in fish (Hodson, 1988). Even though Cu and Zn uptake is biologically controlled, increasing sediment concentrations were moderately correlated with those levels achieved in fish ($r=0.70$ and 0.76).

The Hg tissue concentration reported for stn CS-128 of 0.17 µg/g ww was statistically higher than either that reported for the control and reference sediments, as well as most other test sediments. However, the concentration was no greater than twice the control or reference values. Pre-exposure and control Hg tissue concentrations were 0.06 to 0.09 µg/g and agreed with values found in a previous bioaccumulation laboratory test on Hg-contaminated St. Lawrence River sediments (Bedard and Petro, 1992a).

An examination of site to site differences revealed stn CS-109 most frequently had tissue metal concentrations that differed from reference and test sediment exposures. This occurred for Cu, Pb and Zn. Coincidentally, stn CS-109 also had the highest sediment concentration for this same group of metals. As mentioned earlier, any sediment found internally, within the gastrointestinal tract could be contributing entirely to the whole-organism tissue concentrations.

Concentration factors were calculated to assess the relative availability of each trace metal for the test and reference sediments. The biota-sediment accumulation factor (BSAF) is defined as the ratio of inorganic chemical concentration in the fathead minnow to that in the bulk sediment as determined on the stored samples (Table 9) (Lake *et al.*, 1990). Each individual replicate that was analysed consisted of ten individual animals.

TABLE 11. Spearman rank correlation analysis summary indicating significant negative (inverse) or positive (direct) correlation between sediment bulk metal chemistry and fathead minnow whole-organism bulk metal concentrations for St. Lawrence River 1997 reference and test sediments.

Metal	Correlation Coefficient (r)	Significance Level (p)
Cadmium	- .291	0.47
Copper	.702	0.08
Mercury	.729	0.07
Manganese	.336	0.41
Nickel	.468	0.25
Lead	.864	0.03
Zinc	.763	0.06

Shading indicate significance levels < 0.10; Sample size n=7.

$$BSAF = C_t / C_s$$

where,

C_t = tissue metal contaminant concentration (µg/g tissue, dry weight)
C_s = sediment metal contaminant concentration (µg/g sediment, dry weight)

The resulting BSAFs fell into the following categories:

Biota-Sediment Accumulation Factor	Metal
< 0.1	Manganese, Nickel, Lead
0.1 - 0.5	Cadmium, Copper
1.0	Mercury
>1.0	Zinc

BSAFs > 1.0, indicating that the chemical found in the organism surpassed those levels found in the bulk sediment, were reported at certain test locations for Hg and Zn. Minnow Zn concentrations were 1.6-times to 2.4-times greater than the Zn concentration measured in the sediment at stns CS-128, -156 and -167. The same degree of Zn availability was measured in the reference exposure (BSAF: 2.1) suggesting the chemical is biologically regulated in order to maintain an internal body concentration of roughly 44 µg/g ww. Since Zn is a vital element for normal body function it is not uncommon for the chemical to achieve a constant body burden that surpasses that level measured in the surrounding environment. A similar set of BSAFs have been noted in other sediment bioassays using the fathead minnow in three week laboratory sediment tests (Bedard and Petro, 1992b; 1997a; 1997b; 1997c).

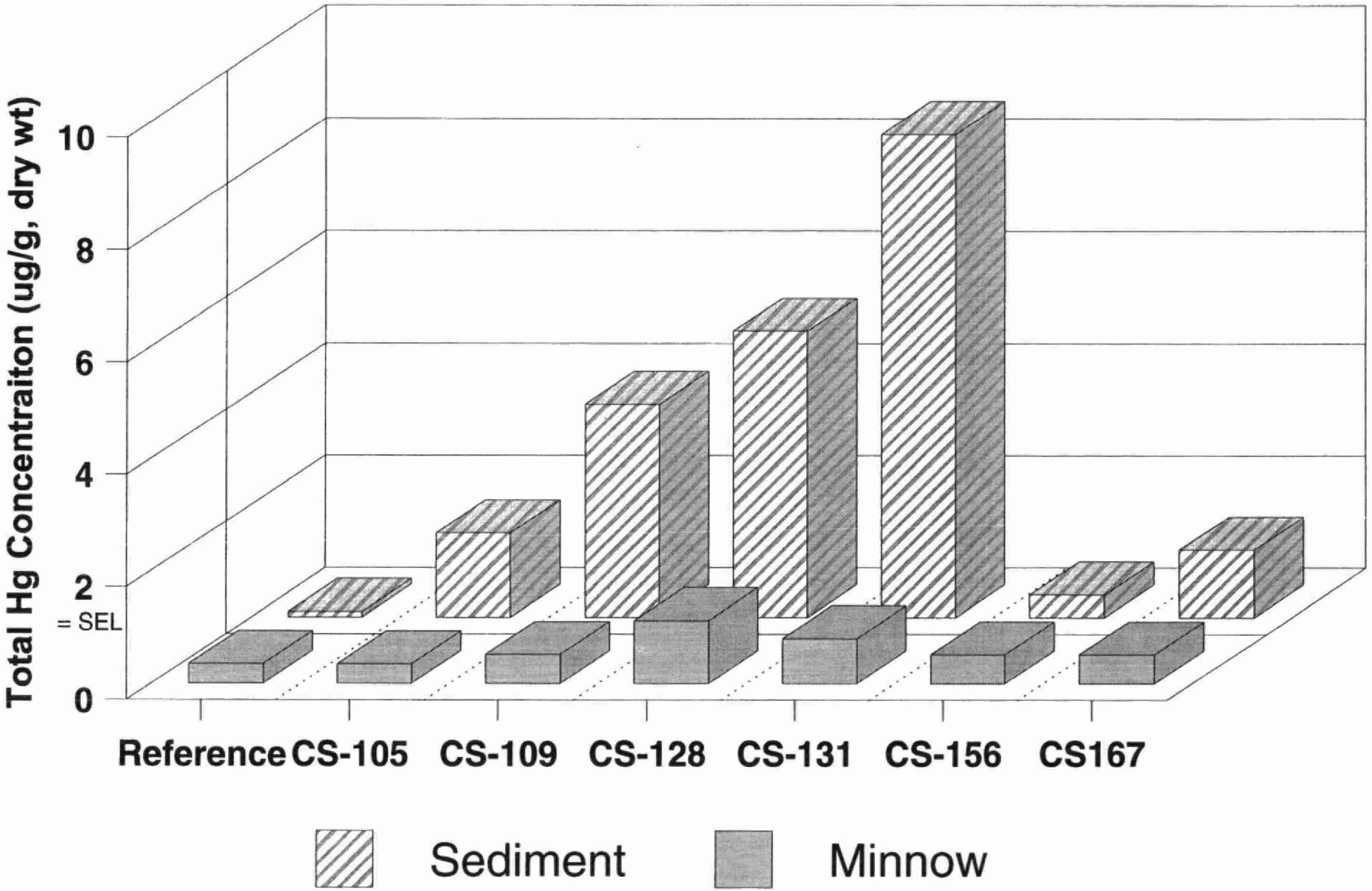
Mercury BSAF's greater than unity was reported only for those less contaminated sites and included stn CS-156 and the reference site (stn CS-175) (Figure 3). The background concentration of Hg in pre-exposed, negative control and reference animals ranged from 0.41 to 0.59 µg/g, dw. The high background Hg tissue concentration, in conjunction with the relatively low Hg concentrations found in 'clean' sediments, resulted in an erroneously high BSAF ratio. This situation also gave rise to a BSAF of 1.3 measured at stn CS-156. Overall, all St. Lawrence River test sediments showed minimal metal availability in the short-term tests.

4.0 DISCUSSION

Spatial Trends in Sediment Toxicity and Mercury Bioaccumulation

Site to site differences in sediment quality were identified in order to measure any spatial trends in biological effect. This was determined by the magnitude of an effect using statistical test methods and total Hg fathead minnow concentrations as they relate to existing federal and provincial tissue guidelines. Each endpoint was considered as being either a significant, toxic

Fig. 3. Total Mercury Concentration in Fathead Minnow and Sediment



(T) or non-significant, non-toxic (N) response (Table 12). In addition, the lethality endpoint received a greater weighting over the respective sublethal endpoint, where applicable. Relative differences among the test sediments was based on the total number of positive hits recorded for each of the five biological endpoints along with the Hg bioaccumulation data.

Station CS-131 sediment was found to be slightly impaired based on the poor rate of growth observed in the *Hexagenia limbata* toxicity test. Among the remaining sites, stn CS-128 was associated with a marginal reduction in mayfly growth that was considered insufficient to warrant a toxic response. Five of the test sediments were deemed of high sediment quality due to the lack of any significant biological effect. The highest minnow Hg concentration was measured for stn CS-128 (0.17 µg/g, wet weight) and was appreciably less than the International Joint Commission (IJC) Aquatic Life Guideline of 0.50 µg/g ww (IJC, 1988). The provincial consumption guideline also employs 0.50 µg/g as a minimum acceptable level for eating restrictions of sportfish (MOEE/MNR, 1997). In addition, Hg availability from this site was relatively low (BSAF=0.21) and did not influence the degree of sediment quality.

Relationships Between Biological Endpoints and Sediment Physical/Chemical Characteristics

Spearman rank correlation coefficients were calculated among each test species and endpoint (Table 13). None of the endpoints were significantly intercorrelated. The absence of any meaningful inter-relationships both within and among bioassays was a result of uniformity among sites e.g. non-toxic.

The toxicity endpoints were also compared to sediment physical, nutrient and chemical parameters to aid in determining potential causes for the observed laboratory effects (Table 14). The analysis pointed out a number of sediment variables that could have influenced the biological data but not to an extent that caused significant differences in toxicity. The strongest negative correlation was found between sediment Hg concentration and mayfly survival rate. Mayfly survival fell within a narrow response range of 90% to 100% survival and occurred over a wide gradient in Hg sediment concentrations, 0.7 µg/g to 6.1 µg/g. There was a weak positive relationship ($r=0.65$; $p=0.10$) between sediment type and mayfly growth, suggesting only a minor influence of sediment nutrient content upon organism growth. The distribution of Cd and Ni concentrations among the reference and test sediments was related to sediment characteristics including particle size (%fines) and organic content (TOC).

Three of the test sediments had chemical concentrations that exceeded the PSQG-SEL for Hg. Chemical concentrations which exceed the SEL are predicted to have a better likelihood of eliciting an adverse biological effect based on field-derived criteria using benthic community structure data (Persaud *et al.*, 1992). The degree to which stns CS-109, -128 and -131 sediment Hg concentration surpassed the SEL of 2 µg/g was by 1.8-fold to 3.0-fold and was not associated with any notable lethal effect in the laboratory toxicity tests. Other studies that examined Hg dynamics between sediment and benthos using similar test concentrations also failed to identify a related acute biological response. For instance, Saouter *et al.*, (1993) reported a 9-Day NOEC concentration of >1.1 µg/g dw for either inorganic or methylated form of Hg based on sublethal data for the mayfly, *Hexagenia rigida.* Similarly, in spiked-sediment exposures at concentrations of 10 µg/g dw for inorganic Hg or 1.0 µg/g for methyl Hg, a 15-day NOEC of > 10 µg/g was derived for mayfly growth and survival (Odin *et al.*, 1994). In a more analogous situation, a series of sediment toxicity tests were conducted in 1990 on Hg-contaminated field sediments from

TABLE 12. Spatial variability in sediment toxicity for St. Lawrence River 1997 samples.

Station	Sediment Total Hg (µg/g dry wt)	Minnow Total Hg (µg/g dry wt)	Mayfly Mortality	Mayfly Ave wt	Midge Mortality	Midge Ave wt	Minnow Mortality
St. Lawrence R. Stn CS - 175	0.1	0.43	N	N	N	N	N
Stn CS - 167	1.0	0.52	N	N	N	N	N
Stn CS - 156	0.7	0.52	N	N	N	N	N
Stn CS - 105	1.5	0.41	N	N	N	N	N
Stn CS - 109	3.6	0.52	N	N	N	N	N
Stn CS - 128	6.1	1.10	N	N	N	N	N
Stn CS - 131	5.4	0.79	N	T	N	N	N

N - Not Toxic, % mortality less than control criteria or $p>0.05$ and $p>0.10$ for growth data;
T - Toxic, % mortality greater than control criteria or $p<0.05$ and $p<0.10$ for growth data.

TABLE 13. Spearman rank correlation coefficients indicating significant positive (direct) correlations among toxicity data for St. Lawrence River 1997 sediments.

	Mayfly Survival	Mayfly Growth	Midge Survival
Mayfly Growth	n.s.		
Midge Survival	n.s.	n.s.	
Midge Growth	n.s.	n.s.	n.s.

* $p < 0.05$; n.s. - Not Significant at $p>0.05$.

TABLE 14. Spearman rank correlation analysis summary indicating significant negative (inverse) or positive (direct) correlation between biological endpoints and sediment physical and chemical parameters for St. Lawrence River 1997 samples.

Toxicity Endpoint	Mayfly Survival	Mayfly Growth	Midge Survival	Midge Growth
Bulk Concentration	- Hg **	+ TOC * + Cd *		- As * - Pb *
TOC Corrected [a]	+ Ni **			

** $p < 0.05$; * $p < 0.10$.

a Included Cd and Ni.

Peninsula Harbour, Ontario (Bedard and Petro, 1992b). The tests involved the same test species, test design and test endpoints as those described herein. Over a range of sediment Hg concentration of 1.4 µg/g to 9.8 µg/g, no significant lethal or sublethal effect was measured. This limited data set suggests Hg was not contributing significantly to a biological effect, at least when it occurs in isolation, as was the case for the 1997 St. Lawrence River test sediments.

Previous sediment toxicity tests conducted at several sites in the St. Lawrence river that match those studied herein, found variable species-specific responses (Bedard and Petro, 1992a). Following identical test methods, the bioassays found sediment collected near stn CS-105 with a Hg concentration in excess of 7.5 µg/g was associated with reductions in mayfly growth as much as 79% and midge growth of 89%. The same sediment also had Cu, Pb and Zn concentrations that surpassed the SEL. In comparison, examination of the 1997 test results reveal sediment from stns CS-128 and CS-131 exerted a sublethal effect of 27% to 47% reduction in body weight but only for one of the benthic species, *Hexagenia limbata*. These test sediments had slightly lower Hg sediment concentrations of 5.4 and 6.1 µg/g and no additional metals were found above the SEL. A possible reason for the higher frequency of significant effects in 1991 could be due to elevated concentrations of multiple metals and the possible presence of other organic contaminants. Previous sediment surveys of the area have indicated relatively high amounts of oils and greases in bottom sediments (Metcalfe-Smith *et al.*, 1995; Richman, 1996). Unfortunately, in 1991, the bioassay sediments along with concurrent field samples were not analyzed for organic contaminants other than PCBs and total PAHs (Richman, 1994; Bedard and Petro, 1992a). There is some indication that some of the test sediments may have contained petroleum substances such as those present in fuel oil or Bunker C oil. Therefore, in 1997 the test sediments were also analyzed for TPHs in an attempt to identify differences in organic contaminant composition.

Some similarities did exist between the 1991 and 1997 studies. Lower sediment Hg concentrations between 0.3 to 2.3 µg/g (Ave: 0.9 µg/g) measured in six of nine test samples in 1991 were associated with no adverse lethal or sublethal biological effect. This agreed with the 1997 findings at four sites with Hg sediment concentrations ranging from 0.7 to 3.5 µg/g (Ave: 1.7 µg/g). In both studies, factors such as substrate type and nutrient enrichment was ruled out as having an important influence on the outcome of the biological responses.

Importance of Mercury Bioavailability and Bioaccumulation

Mercury is one of the few inorganic compounds that is highly persistent in the environment and capable of becoming biomagnified throughout the aquatic food chain (NRCC, 1979; OMOE, 1992). Biomagnification is a process that describes the transfer of a contaminant in biota due to predator-prey interactions, such that the residue concentration of Hg increases from one trophic level to the next (Rand and Petrocelli, 1985). Contaminated sediment is an originating source of Hg into the aquatic food chain through its' uptake by bottom-dwelling organisms (Jackson, 1988; Dukerschein *et al.*, 1992; Parkman and Meili, 1993; Beauvais *et al.*, 1995). The fathead minnow is a benthic species that forages in sediment and when used in static laboratory tests can represent the degree to which Hg can bioaccumulate in a worst-case situation. Fish can acquire contaminants that are sorbed to sediment through the direct ingestion of sediment particles, as well as by contact with particulate and dissolved forms of the chemical that may be present in the water-column (Rudd *et al.*, 1983; Dabrowska *et al.*, 1996; Kannan *et al.*, 1998). Sites of Hg uptake in fish include the gut through ingestion, as well as the gills during respiration (Boudou *et*

al., 1991). Both sources can behave in an additive manner in determining fish Hg tissue concentrations (Turner and Swick, 1983).

Mercury uptake by biota will vary depending on a number of biotic and abiotic factors. Mercury levels in fish have been shown to preferentially occur in the methylated or organic form, CH_3Hg^+, as compared to the elemental or inorganic form, Hg^{+2} (Huckabee *et al.*, 1979; Rudd *et al.*, 1983; Grieb *et al.*, 1990; Francesoni and Lenanton, 1992). In constrast, MeHg constitutes a very small fraction (<0.1%) of the total Hg in the aquatic environment (D'Itri, 1990). The top surficial layer (1-2 cm) of bedded sediment is considered to have somewhat higher amounts of MeHg relative to the amount associated with deeper sediment layers as a result of enhanced microbial methylation (Rudd *et al.*, 1983). Increased oxygenation at the sediment surface also promotes the mobilization of sulfide-bound Hg making it more available for methylation (D'Itri, 1990). Methyl Hg constituted 0.3% to 2.5% of the total Hg in St. Lawrence River sediments and averaged 1.0% (n=17) (Richman, 1994).

The concentration of Hg in fish is also a function of the concentration of MeHg found in the water column (Jackson, 1991). The foraging and swimming activity of fathead minnows in the sediment bioassay test chambers resulted in a resuspension of sediment particles into the water-column. Bioturbation changes the concentration of total Hg in the overlying water and also affects Hg speciation. The transfer of Hg from sediment to water, either dissolved or particulate-bound, appears to favor the methylated form of Hg (Saouter *et al.*, 1991; Burgess and Scott, 1992; Saouter *et al.*, 1993). Fish can readily assimilate even small quantities of MeHg present in sediment or water which enhances Hg bioaccumulation (D'Itri, 1990; Boudou *et al.*, 1991). Once accumulated, Hg is depurated at very slow rates and in long-lived fish species will lead to Hg biomagnification. Benthic invertebrates typically have a lower percentage of MeHg relative to fish species (Huckabee *et al.*, 1979; Jackson, 1988; Francesoni and Lemanton, 1992). These differences are partially explained by the additional internal methylation capacity in fish due to the presence of microbial organisms in fish intestine (Rudd *et al.*, 1983). Ameliorating effects towards MeHg uptake include microbial demethylation and increased affinity to sulphide under aerobic conditions (Jackson, 1991). Improved sediment oxygenation also renders humic-bound Hg less available (D'Itri, 1990). The above relationships need to be recognized as potential contributing factors in Hg uptake.

Despite the constantly changing inorganic:organic Hg exposure concentrations during testing, the Hg uptake response time by fish is relatively short. Several laboratory studies have demonstrated Hg can reach a steady-state equilibrium between organism and sediment-water exposures in a relatively short period of time. Time to steady-state was achieved in 10 to 15 days using the mayfly, *Hexagenia rigida* (Saouter *et al.*, 1991) and 10-days using guppies (Kudo and Mortimor, 1979). The test duration used in the fathead minnow bioaccumulation assay should provide a reasonable approximation of steady-state Hg tissue levels. Also the absence of any fish mortality suggests the animals had a high tolerance towards the test sediments which should increase exposure to any sediment-associated or water-borne contaminants. Negligible fish avoidance increases the level of contact with the sediment when the fish are actively feeding near the sediment surface. For these reasons, the minnow test is expected to provide information describing relative Hg availability along a gradient of sediment contamination (Lester and McIntosh, 1994; Harkey et al., 1994).

After 21 days, the surviving fathead minnows achieved whole-body Hg tissue residues that statistically differed from the control animals at a single site (stn CS-128). The average body

residue was 0.17 µg/g ww and represented less than a two-fold increase to concentrations attained through culturing practices alone (0.09 µg/g). In addition, the corresponding BSAF of 0.2 is indicative of low Hg availability and was not unlike that attained at other test sites. Overall, the BSAFs ranged from 0.13 to 1.3 calculated on a dry weight basis. In comparison, the reference exposure (stn CS-175) resulted in the highest BSAF of 4.3, which is not truly indicative of increased Hg availability but rather an artifact due to high Hg background tissue levels.

There is proof of high Hg background concentrations existing in batches of control fish exposed to clean water or sediment. For instance, guppies exposed for two months in a sediment and water system or for 125 days in water only resulted in a Hg body burden of 1.1 µg/g (Gillespie and Scott, 1971; Gillespie, 1972). There are several reasons that could help explain this outcome. Parks *et al.*, (1989) suggests that in clean sediments there is a concentration gradient between MeHg in overlying water relative to that associated with the sediment pore-water that differs from that found in Hg-contaminated sediment. It is the steepness in the chemical concentration gradient that drives the rate of Hg methylation and subsequent biological uptake. A lower concentration gradient associated with clean conditions can lead to increased Hg methylation in water, as compared to a lower methylation rate for more contaminated conditions. The resulting high Hg concentration measured in the negative control and reference minnows will introduce a certain degree of interference in detecting any notable differences in Hg tissue concentrations among the test sediments. Furthermore, it has been demonstrated that the inherent presence of bacterial flora in fish gut will enhance MeHg production and making uptake independent of differences in Hg exposure concentrations (Rudd *et al.*, 1983).

A direct comparison was made between fathead minnow Hg tissue concentrations obtained in 1997 to those reported in an earlier 1991 study on St. Lawrence River sediments (Bedard and Petro, 1992a). Of the original nine sites in 1991, three of the sites were re-evaluated in 1997 but not necessarily at the exact same sampling location. Despite any variability in sampling procedures, for the most part there was little or no temporal differences in fathead minnow Hg body burdens. The average Hg fish concentration in 1991 was 0.10 µg/g ± 0.03 ww based on a sample size of eight (Bedard and Petro, 1992a). Similarly, in 1997 the average minnow Hg concentration was 0.09 µg/g ± 0.04 ww for seven sites (Table 9). No apparent change in final tissue Hg concentrations were found between the two sampling periods. Mercury concentrations in negative control minnows was 0.06 µg/g and 0.08 µg/g ww in 1991 and 1997, respectively. In terms of Hg availability, the 1991 study did report a higher number of BSAFs exceeding 1.0 (Range: 0.15 to 3.1) even for those sites with relative high Hg sediment concentrations. The range of BSAFs observed for fathead minnows in test sediment in 1997 was 0.09 to 1.3 and coincided with those cited for guppies of 0.4 (Gillespie and Scott, 1971) and 1.3 (Kudo and Mortimor, 1979).

A further comparison was made between final whole-organism total Hg tissue concentrations and existing federal tissue guidelines. The IJC developed guidelines for persistent substances in order to protect against chemical transfer through the aquatic food chain (IJC, 1988). The guideline is based on whole-fish and has been applied to whole-organism young-of-the-year forage fish (Suns *et al.*, 1991). The Hg body burdens measured in the laboratory-exposed fathead minnows reached levels that were more than two-fold lower than the Hg criteria of 0.50 µg/g ww for all of the test sites.

The laboratory bioaccumulation data was also compared with available field data in order to assess to what extent the laboratory results predicted Hg dynamics occurring *in-situ.* Since

1979 there has been periodic monitoring of spottail shiners, *Nostromis sp.* for bioaccumulative substances including Hg. Data has been collected for sites just upstream and downstream of Courtaulds Fibres, downstream of Domtar at the Cornwall marina, and off the northwest portion of Pilon Island near Gray's creek (Hitchin, 1997). Chemical analysis from the most recent collections, 1990 - 1996, report values ranging from 0.02 µg/g, 0.03 µg/g and 0.06 µg/g ww. In comparison, an upstream collection at MacDonnell Island indicated an average background Hg concentration of 0.05 µg/g for 1990 to 1993. The downstream locations appear to have comparable if not lower Hg tissue levels than that found in the upstream reference site. Similarly, the Hg concentration in the bioassay reference animals (stn CS-175) of 0.06 µg/g ww also matched those at the test sites, despite the low Hg sediment concentration of 0.1 µg/g dw. Overall, the laboratory-derived numbers were not unlike those found in field-collected forage fish.

A certain degree of Hg can be associated with fish gut on a whole-organism basis for clean and contaminated sites. Since the Hg analyses was based on unpurged animals, the entire Hg body residue in the bioassay fish could have been derived solely from sediment-associated Hg that was contained within the gut of the organism, assuming a gut content of 20%. This applied even at those sites which resulted in significantly higher Hg fathead minnow concentrations e.g. 0.12 µg/g ww; Stn CS-131 and 0.17 µg/g ww; Stn CS-128.

Since Hg is known to be highly bioaccumulative it is important to study trends in other trophic levels. Mercury analyses on benthic organisms obtained from several St. Lawrence River sites in the vicinity of Courtaulds Fibres had a mean Hg concentration of 0.02 µg/g ww with a maximum value of 0.06 µg/g (Richman, 1994). The concentrations observed in field benthos overlapped those found in local native forage fish (0.02 µg/g to 0.06 µg/g) (Hitchin, 1997). Somewhat higher Hg tissue concentrations were measured in fathead minnows in the 1997 laboratory tests (0.06 µg/g to 0.17 µg/g). Bottom-dwelling fish represent a link in the aquatic food chain and appear to have intermediate contaminant levels. The extent to which Hg is biomagnified is best represented by long-lived fish species. In 1994, top predator sportfish Hg contaminant levels for northern pike (55 cm) and walleye (50 cm) averaged 0.375 µg/g and 0.500 µg/g ww (Dreier *et al.*, 1997). There appears to be a progressive increase in total Hg biota concentrations of at least an order of magnitude between different trophic levels.

5.0 CONCLUSIONS

Laboratory sediment toxicity tests conducted in 1997 on St. Lawrence River surficial sediments found few toxicological effects among sites. Mayfly growth was the most sensitive endpoint and identified stn CS-131 as being slightly impaired. *Hexagenia* nymphs were 47% smaller than the group of reference animals. Each test sediment did not negatively affect organism survival for any of the test species. Acceptable levels of midge growth were also found among test, reference and control sediments.

Chemical and physical characterization of the test sediments indicated total mercury concentrations in exceedence of the PSQG-Severe Effect Level concentration for three of the seven test samples. Sediment Hg concentrations of 3.6 µg/g to 6.1 µg/g occurred for stns CS-109, CS-128 and CS-131. Most of the remaining trace metals were measured at levels representative of Lowest Effect Level concentrations. Organic chemical analysis indicated low-level concentrations for total polychlorinated biphenyls (PCBs), total polycyclic aromatic hydrocarbons (PAHs) and total petroleum hydrocarbons (TPHs).

Correlation analysis of the biological, physical and chemical data failed to find any important inter-relationships. This was probably due to the narrow biological range in response, in conjunction with a relatively small sample size.

The fathead minnow short-term bioaccumulation test illustrated a low availability of total Hg under controlled laboratory conditions using static exposures. Fathead minnow Hg tissue concentrations on unpurged animals were within twice the concentration found in the reference fish. Mercury body burdens were well below the International Joint Commission Aquatic Life Guideline of 500 ng/g ww. Although Hg availability and uptake was marginal and could be accounted solely by the process of sediment ingestion, there was a slight correlation between total Hg concentrations measured in the sediment to those measured in the fish. The transfer of Hg-contaminated sediment into bottom-dwelling fish could be ecologically relevant given the capacity of Hg to become biomagnified in the aquatic food chain. Mercury concentrations in laboratory-exposed minnows were similar to those detected *in-situ* according to the analysis of indigenous spottail shiners.

REFERENCES

American Society for Testing and Materials (ASTM). 1997a. *Standard Test Methods for Measuring the Toxicity of Sediment-associated Contaminants with Fresh Water Invertebrates.* E1706-95b. In Annual Book of ASTM Standards, Vol. 11.05. Philadelphia, PA. pp. 1138-1220.

American Society for Testing and Materials (ASTM). 1997b. *Guide for Collection, Storage, Characterization, and Manipulation of Sediments for Toxicological Testing.* E1391-94. In Annual Book of ASTM Standards, Vol. 11.05. Philadelphia, PA. pp. 767-787.

Anderson, J. 1990. *St. Lawrence River Environmental Investigations. Volume 4. Assessment of Water and Sediment Quality in the Cornwall Area of the St. Lawrence River, 1985.* Ontario Ministry of Environment, Water Resources Branch, ISBN 0-7729-7562, Toronto, Ontario. 39 p.

Ankley, G.T. 1996. Evaluation of Metal/Acid-volatile Sulfide Relationship in the Prediction of Metal Bioaccumulation by Benthic Macroinvertebrates. *Environ. Toxicol. Chem.* 15:2138-2146.

Beauvais, S.L., J.G. Wiener and G.J. Atchison. 1995. Cadmium and Mercury in Sediment and Burrowing Mayfly Nymphs (*Hexagenia*) in the Upper Mississippi River, USA. *Arch. Environ. Contam. Toxicol.* 28:178-183.

Bedard, D. and S. Petro. 1992a. *St. Lawrence River/Cornwall Study 1991 Laboratory Sediment Bioassay Report,* Ontario Ministry of Environment, Water Resources Branch, Internal Technical Report, Toronto, Ontario. 17 p.

Bedard, D. and S. Petro. 1992b. *Peninsula Harbour 1990 Laboratory Sediment Bioassay Report,* Ontario Ministry of Environment, Water Resources Branch, Internal Technical Report, Toronto, Ontario. 14 p.

Bedard, D. and S. Petro. 1997a. *Report on Laboratory Sediment Bioassay Results for Fly Creek Stormwater Retention Pond Sediments in Cornwall, Ontario 1995,* Ontario Ministry of Environment, Standard Development Branch, Internal Technical Report, Toronto, Ontario. 19 p.

Bedard, D. and S. Petro. 1997b. *Report on Laboratory Sediment Bioassay Results for Oshawa Second Marsh Sediments 1995*, Ontario Ministry of Environment, Standards Development Branch, Internal Technical Report, Toronto, Ontario. 18 p.

Bedard, D. and S. Petro. 1997c. *Laboratory Sediment Bioassay Report on St. Marys River Sediments 1992 & 1995*, Ontario Ministry of Environment and Energy, Standards Development Branch, ISBN 0-7778-6674-9, Toronto, Ontario. 59 p.

Bedard, D., A. Hayton and D. Persaud. 1992. *Ontario Ministry of the Environment Laboratory Sediment Biological Testing Protocol*, Ontario Ministry of the Environment, Water Resources Branch, ISBN 0-7729-9924-4, Toronto, Ontario. 23 p.

Boese, B.L., H. Lee II, D.T. Specht, J. Pelletier and R. Randall. 1996. Evaluation of PCB and Hexachlorobenzene Biota-Sediment Accumulation Factors Based on Ingested Sediment in a Deposit-feeding Clam. *Environ. Toxicol. Chem.* 15:1584-1589.

Boudou, A., M. Delnomdedieu, D. Georgescauld, F. Ribeyre and E. Saouter. 1991. Fundamental Roles of Biological Barriers in Mercury Accumulation and Transfer in Freshwater Ecosystem (analysis at organism, organ, cell and molecular levels). *Water, Air, and Soil Pollution.* 56:807-821.

Borgmann, U., W.P. Norwood and C. Clarke. 1993. Accumulation, Regulation and Toxicity of Copper, Zinc, Lead and Mercury in *Hyalella azteca. Hydrobiologia* 259:79-89.

Borgmann, U. and W.P. Norwood. 1997. Toxicity and Accumulation of Zinc and Copper in *Hyalella azteca* Exposed to Metal-spiked Sediments. *Can. J. Fish. Aquat. Sci.* 54:1046-1054.

Burgess, R.M. and K.J. Scott. 1992. The Significance of In-Place Contaminated Marine Sediments on the Water Column:Processes and Effects. In: G.A. Burton Jr., (Ed.) *Sediment Toxicity Assessment*, Lewis Publishers, Ann Arbor, Michigan, pp.129-165.

Campbell, P.G.C., A.G. Lewis, P.M. Chapman, A.A. Crowder, W.K. Fletcher, B. Imber, S.N. Luoma, P.M. Stokes and M. Winfrey. 1988. *Biologically Available Metals in Sediments.* National Research Council of Canada, NRCC No. 27694, Ottawa, Ontario. 298 p.

Dabrowska, H., S.W. Fisher, K. Dabrowski and A.E. Staubus. 1996. Dietary Uptake Efficiency of HCBP in Channel Catfish: the Effect of Fish Contaminant Body Burden. *Environ. Toxicol. Chem.* 15:746-749.

D'Itri, F.M. 1990. The Biomethylation and Cycling of Selected Metals and Metalloids in Aquatic Sediments. In: R. Baudo, J.P. Giesy, H. Muntau, (Eds.) *Sediments: Chemistry and Toxicity of In-Place Pollutants*, Lewis Publishers, Ann Arbor, Michigan, pp.163-214.

Dreier, S.I., J. Anderson, J. Biberhofer, M. Eckersley, R. Helliar, M.B.C. Hickey, L. Richman, F. Stride and St. Lawrence River (Cornwall) Remedial Action Plan Public Advisory Commitee. 1997. *Great Lakes, Great River Remedial Action Plan for St. Lawrence River (Cornwall) Area of Concern - Stage 2 Report: The Recommended Plan.* November 1997, ISBN 0-7778-6881-4. 203 p.

Dukerschein, J.T., J.G. Wiener, R.G. Rada and M.T. Steingraeber. 1992. Cadmium and Mercury in Emergent Mayflies (*Hexagenia bilineata*) from the Upper Mississippi River. *Arch. Environ. Contam. Toxicol.* 23:109-116.

Environment Canada (EC). 1994. *Guidance Document on Collection and Preparation of Sediments for Physicochemical Characterization and Biological Testing*, Environmental Protection Service, Report EPS 1/RM/29, Ottawa, Ontario. 144 p.

Franscesconi, K.A. and R.C.J. Lenanton. 1992. Mercury Contamination in a Semi-enclosed Marine Embayment: Organic and Inorganic Mercury Content of Biota, and Factors Influencing Mercury Levels in Fish. *Marine Environ. Res.* 33:189-212.

Friesen, M.K. 1981. Hexagenia rigida (McDunnough). In: S.G. Lawrence (Ed.), *Manual for the Culture of Selected Freshwater Invertebrates*, Can. Spec. Publ. Fish. Aquat. Sci., 54 : 127-142.

Gillespie, D.C. 1972. Mobilization of Mercury from Sediments into Guppies. *J. Fish. Res. Bd*

Can. 29:1035-1041.

Gillespie, D.C. and D.P. Scott. 1971. Mobilization of Mercuric Sulphide from Sediment into Fish under Aerobic Conditions. *J. Fish. Res. Bd. Can.* 28:1807.

Grieb, T.M., C.T. Driscoll, S.P. Gloss, C.L. Schofield, G.L. Bowie and D.B. Porcella. 1990. Factors Affecting Mercury Accumulation in Fish in the Upper Michigan Peninsula. *Environ. Toxicol. Chem.* 9:919-930.

Harkey, G.A., M.J. Lydy, J. Kukkonen and P.F. Landrum. 1994. Feeding Selectivity and Assimilation of PAH and PCB in *Diporeia* spp.. *Environ. Toxicol. Chem.* 13:1445-1455.

Hitchin, G. 1997. *St. Lawrence River Organochlorine and Mercury Residues in YOY Spottail Shiners 1979-1996.* Internal Technical Memorandum. Ministry of Environment, Environmental Monitoring and Reporting Branch. 3 p.

Hodson, P.V. 1988. The Effect of Metal Metabolism on Uptake, Disposition and Toxicity in Fish. *Aquatic Toxicology.* 11:3-18.

Huckabee, J.W., J. W. Elwood and S.G. Hildebrand. 1979. Accumulation of Mercury in Freshwater Biota. In: J.O. Nriagu (Ed.) *The Biogeochemistry of Mercury in the Environment,* Elsevier/North-Holland Bomedical Press, Amsterdam, The Netherlands. pp.276-301.

International Joint Commission (IJC). 1988. Revised Great Lakes Water Quality Agreement of 1978. January 1988.

Jaagumagi, R. and D. Persaud. 1996. *An Integrated Approach to the Evaluation and Management of Contaminated Sediments.* Ontario Ministry of Environment and Energy, Standards Development Branch, ISBN 0-7778-3845-1, Toronto, Ontario. 50 p.

Jackson, T.A. 1988. Accumulation of Mercury by Plankton and Benthic Invertebrates in Riverine Lakes of Northern Manitoba (Canada): Importance of Regionally and Seasonally Varying Environmental Factors. *Can. J. Fish. Aquat. Sci.* 45:1744-1757.

Jackson, T.A. 1991. Biological and Environmental Control of Mercury Accumulation by Fish in Lakes and Reservoirs of Northern Manitoba, Canada. *Can. J. Fish. Aquat. Sci.* 48:2449-2470.

Kannan, K., R.G. Smith Jr., R.F. Lee, H.L. Windom, P.T. Heitmuller, J.M. Macauley and J.K. Summers. 1998. Distribution of Total Mercury and Methyl Mercury in Water, Sediment, and Fish from South Florida Estuaries. *Acrh. Environ. Contam. Toxicol.* 34:109-118.

Kauss, P., Y.S. Hamdy and B.S. Hamma. 1988. *St. Lawrence River Environmental Investigations. Volume 1. Background: Assessment of Water, Sediment and Biota in the Cornwall, Ontario and Massena, New York Section of the St. Lawrence River 1979-1982.* Ontario Ministry of Environment, Water Resources Branch, ISBN 0-7729-3559-9, Toronto, Ontario. 157 p.

Kudo, A. and D.C. Mortimer. 1979. Pathways for Mercury Uptake by Fish from Bed Sediments. *Environ. Pollut.* 19:239-245.

Lake, J.L., N.I. Rubinstein, H. Lee II, C.A. Lake, J. Heltshe and S. Pavignano. 1990. Equilibrium Partitioning and Bioaccumulation of Sediment-associated Contaminants by Infaunal Organisms. *Environ. Toxicol. Chem.* 9 : 1095-1106.

Lester, D.C. and A. McIntosh. 1994. Accumulation of Polychlorinated Biphenyl Congeners from Lake Champlain Sediment by *Mysis relicata. Environ. Toxicol. Chem.* 13:1825-1841.

Luoma, S. 1983. Bioavailability of Trace Metals to Aquatic Organisms - A Review. *Sci. Total Environ.* 28:1-22.

Metcalfe-Smith, J., L. Richman, R. Santiago, J. Biberhofer and J. Anderson. 1995. *Chemical Characterization of St. Lawrence River Sediments in the Vicinity of the Oil Tank Storage Area, Cornwall, Ontario.* St. Lawrence River RAP Technical Report No. 6. Kingston, Ontario.17 p..

Millar, C.E., L.M. Turk and H.D. Foth. 1965. *Fundamentals of Soil Science*, 9th Ed., John Wiley and Sons, New York, NY.

Mosher, R.G., R.A. Kimerle and W.J. Adams. 1982. *MIC Environmental Assessment Method for Conducting 14-day Partial Life Cycle Flow-through and Static Sediment Exposure Toxicity Tests with the Midge Chironomus tentans*, Report No. E5-82-M-10, Monsanto, St. Louis.

National Research Council of Canada (NRCC). 1979. *Effects of Mercury in the Canadian Environment.* NRCC, Associate Committee on Scientific Criteria for Environmental Quality. NRCC Publication No. 16739. 290 p.

Odin, M., A. Feurtet-Mazel, F. Ribeyre and A. Boudou. 1994. Actions and Interactions of Temperature, pH and Photoperiod on Mercury Bioaccumulation by Nymphs of the Burrowing Mayfly *Hexagenia rigida*, from the Sediment Contamination Source. *Environ. Toxicol. Chem.* 13:1291-1302.

Ontario Ministry of the Environment (OMOE). 1983. *Handbook of Analytical Methods for Environmental Samples.* Vol. 1 and 2. Laboratory Services and Applied Research Branch, Toronto, Ontario.

OMOE. 1989. *A Guide to the Collection and Submission of Samples for Laboratory Analysis.* Laboratory Services Branch, Toronto, Ontario. 81 p.

OMOE. 1992. *Candidate Substances List for Bans or Phase-outs.* Ontario Ministry of Environment, Hazardous Contaminants and Water Resources Branch, Toronto, Ontario. 63 p.

OMOEE. 1994a. *The Determination of Trace Metals in Sediments by Spectro Inductively Coupled Plasma-Optical Emission Spectrometer (ICP-OES).* HMARSOIL-E3062A. Laboratory Services Branch, Etobicoke, Ontario. March 28, 1994. 45 p.

OMOEE. 1994b. *The Determination of Polychlorinated Biphenyls (PCBs), Organochlorines (OCs), and Chlorobenzenes (CBs) in Soil and Sediments by Gas Liquid Chromatography-Electron Capture Detection (GLC-ED).* PSAOC-E3270A. Laboratory Services Branch, Etobicoke, Ontario. July 18, 1994. 37 p.

OMOEE. 1994c. *Water Management Policies, Guidelines, Provincial Water Quality Objectives of the Ministry of Environment and Energy,* Toronto, Ontario. 30 p.

OMOEE. 1995a. *The Determination of Total Carbonate-Carbon in Soil and Sediments by Coulometry.* CARBONAT-E3012A. Laboratory Services Branch, Etobicoke, Ontario.

OMOEE. 1995b. *The Determination of Particle Size on Sediments by MICROTRAC Particle Size Analyzer Model 7991-2.* SMPART-E3128A. Laboratory Services Branch, Etobicoke, Ontario.

OMOEE. 1996. *The Determination of Heavy Metals in Biomaterials by Atomic Absorption Spectrophotometer (AAS).* Laboratory Services Branch, Etobicoke, Ontario.

OMOEE and Ontario Ministry of Natural Resources (OMOEE/OMNR). 1997. *Guide to Eating Ontario Sport Fish 1997-1998.* 19th Edition. ISSN 0826-9653. Toronto, Ontario. 173 p.

Parkman, H. and M. Meili. 1993. Mercury in Macroinvertebrates from Swedish Forest Lakes: Influence of Lake Type, Habitat, Life Cycle, and Food Quality. *Can. J. Fish. Aquat. Sci.,* 50: 521-534.

Parks, J.W., A. Lutz and J.A. Sutton. 1989. Water Column Methylmercury in the Wabigoon/English River-Lake System: Factors Controlling Concentrations, Speciation, and Net Production. *Can. J. Fish. Aquat. Sci.* 46:2184-2202.

Persaud, D., R. Jaagumagi and A. Hayton. 1992. *Guidelines for the Protection and Management of Aquatic Sediment Quality in Ontario.* Ontario Ministry of the Environment, Toronto. 30 p.

Rand, G.M. and S.R. Petrocelli. 1985. *Introduction in Fundamentals of Aquatic Toxicology: Methods and Applications.* Hemisphere Publishing Corp., Washington, 666 p.

Richman, L. 1994. *St. Lawrence River Sediment and Biological Assessment 1991.* Ontario Ministry of Environment and Energy, Environmental Monitoring and Reporting Branch, ISBN 0-7778-2743-3, Toronto, Ontario. 52 p.

Richman, L. 1996. *St. Lawrence River Sediment Assessment 1994, Cornwall, Ontario.* St. Lawrence River RAP Technical Report No. 7. ISBN 0-7778-5652-2, Kingston, Ontario. 61 p.

Richman, L. 1997. *Project Description - St. Lawrence River Sediment Survey, 1997.* Ontario Ministry of Environment and Energy, Toronto, Ontario. 3 p.

Richman, L. and H. Biberhofer. 1998. In Preparation.

Rudd, J.W.M., M.A. Turner, A. Furutani, A.L. Swick and B.E. Townsend. 1983. The English-Wabigon River System: I. A Synthesis of Recent Research with a View towards Mercury Amelioration. *Can. J. Fish. Aquat. Sci.,* 40:2206-2217.

Rukavina, N. 1996. *Site Selection and GIS Analysis of Contaminated Sediment Distribution and Volume for the Proposed Courtaulds' Dredge Site, Cornwall, Ontario.* NWRI Contribution No. 96-204. 13 p.

Rukavina, N. 1998. *Summary of Physical Properties of St. Lawrence River Sediments at Cornwall, Ontario.* NWRI Contribution No. 97-212. 10 p.

Saouter, E., F. Ribeyre and A. Boudou. 1991. Synthesis of Mercury Contamination Mechanisms of a Burrowing Mayfly (*Hexagenia rigida*): Methodological Bases and Principle Results In: J.-P. Vernet (Ed.) *Heavy Metals in the Environment*, Elsevier, Amsterdam, The Netherlands, pp. 175-190.

Saouter, E., L. Hare, P.G.C. Campbell, A. Boudou and F. Ribeyre. 1993. Mercury Accumulation in the Burrowing Mayfly *Hexagenia rigida* (Ephemeroptera) Exposed to CH_3HgCl or $HgCl_2$ in Water and Sediment. *Water Res.* 27:1041-1048.

SAS. 1985. Statistical Analysis Systems, *SAS/STAT User's Guide*, Version 6.03 Edition, SAS Institute, Cary, S.C.

Schubauer-Berigan, M.K., B.D. Monson, C.W. West and G.T. Ankley. 1995. Influence of pH on the Toxicity of Ammonia to *Chironomus tentans* and *Lumbriculus variegatus. Environ. Toxicol. Chem.* 14:713-717.

St. Lawrence RAP Team. 1992. *Remedial Action Plan for the Cornwall-Lake St. Francis Area. Stage 1 Report: Environmental Conditions and Problem Definitions.* Environment Canada & Environment Ontario. ISBN 0-662-19958-8.

Steel, R.G.D. and J.H. Torrie. 1960. *Principles and Procedures of Statistics.* McGraw-Hill Book Company Inc., New York, 481 p.

Stephan, C.E. 1977. Methods for Calculating an LC50, In: F.L. Mayer and J.L. Hamelink (Eds.) *Aquatic Toxicology and Hazard Evaluation*, ASTM STP 634, Philadelphia PA, pp.65-84.

Suns, K., G. Hitchin and D. Toner. 1991. *Spatial and Temporal Trends of Organochlorine Contaminants in Spottail Shiners (Notropis hudsonius) from the Great Lakes and their Connecting Channels (1975-1988).* Ministry of the Environment, ISBN 0-7729-8411-5, Toronto, Ontario. 97 p.

Thurston, R.V., R.C. Russo and G.R. Phillips. 1983. Acute Toxicity of Ammonia to Fathead Minnows. *Trans. American Fisheries Society* 112:705-711.

Thurston, R.V., R.C. Russo, E.L. Meyn, R.K. Zajdel and C.E. Smith. 1986. Chronic Toxicity of Ammonia to Fathead Minnows. *Trans. American Fish. Soc.* 115:196-207.

Townsend, B.E., S.G. Lawrence and J.F. Flannagan. 1981. Chironomus tentans (Fabricius). In: S.G. Lawrence (Ed.), *Manual for the Culture of Selected Freshwater Invertebrates*, Can. Spec. Publ. Fish. Aquat. Sci., Vol. 54, pp. 127-142.

Turner, M.A. and A.L. Swick. 1983. The English-Wabigon River System: IV. Interaction between Mercury and Selenium Accumulated from Waterborne and Dietary Sources by Northern Pike *(Esox lucius). Can. J. Fish. Aquat. Sci.,* 40:2241-2250.

U.S. Environmental Protection Agency (US EPA). 1987. *Guidelines for the Culturing of Fathead*

Minnows Pimephales promelas for use in Toxicity Tests. EPA-600-3-87-001. Environmental Research Laboratory, Duluth, MN, 42 pp.

Whiteman, F.W., G.T. Ankley, M.D. Kahl, D.M. Rau and MD. Balcer. 1996. Evaluation of Interstitial Water as a Route of Exposure for Ammonia in Sediment Tests with Benthic Macroinvertebrates. *Environ. Toxicol. Chem.* 15:794-801.

(7663)
MOE/LAB/AJHN

MOE/LAB/AJHN
Bedard, Donna
Laboratory sediment
bioassay report on ajhn
Cornwall, 1997 c.1 a aa